CYCLANILIPROLE (296)

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EXPLANATION

Cyclaniliprole was scheduled for residue evaluation as a new compound by the 2017 JMPR at the 48th session of the CCPR (2016).

Cyclaniliprole is an insecticide belonging to the chemical class of diamide insecticides and pyrazole insecticides. Despite its structural similarity to some of the phenylpyrazole insecticides, this substance has a different mode of action, which it shares with other diamide insecticides. Diamides act at the ryanodine receptor, which is critical for muscle contraction.

The Meeting received information from the manufacturer on identity, metabolism, storage stability, residue analysis, use patterns, residues resulting from supervised trials on apple, pear, cherry, plum, peach, apricot, nectarine, grapes, head cabbages, Brussels sprouts, broccoli, cauliflower, cucumber, summer squash, melon, tomato, pepper, lettuces, spinach, mustard greens, kale, soybeans, almond, pecan, tea, fate of residue during processing, and livestock feeding studies.

IDENTITY

ISO common name:	Cyclaniliprole
Chemical name	
IUPAC:	2',3-dibromo-4'-chloro-1-(3-chloro-2-pyridyl)-6'-{[(1*RS*)-1-cyclopropylethyl]carbamoyl}pyrazole-5-carboxanilide
CAS:	3-bromo-*N*-[2-bromo-4-chloro-6-[[(1-cyclopropylethyl)amino]-carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide
CAS Registry No:	1031756-98-5
CIPAC No:	Not allocated at the time of evaluation
Synonyms and trade names:	IKI-3106 (development code number)
Structural formula:	include R and S isomer
Molecular formula:	$C_{21}H_{17}Br_2Cl_2N_5O_2$
Molecular weight:	602.1 g/mol

Cyclaniliprole is a racemic mixture (R and S enantiomers present in a 50:50 w/w ratio). The biological activity of the racemate is due to both enantiomers, which give equal insecticidal activity.

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient (cyclaniliprole)

Parameter	Result	References	Guidelines/method
Appearance:	purity: 99.18% w/w odourless white powder at 20 and 25 °C	[Turner 2012a, report no. JSM0235]	US EPA OPPTS 830.6302 and 830.6303 visual, Munsell colour system (N9.25/84.2% R)
Vapour pressure:	purity: 99.18% w/w 2.4x 10^{-6} Pa at 25 °C 1.65 x 10^{-6} Pa at 20 °C	[Turner 2012b, report no. JSM0241]	OECD 104; Vapour Pressure Balance
Melting point:	purity: 99.18% w/w	[Turner 2012a, report no.	OECD 102;

Parameter	Result	References	Guidelines/method
	241-244 °C	JSM0235]	Metal Block Method
Octanol/water partition coefficient:	purity: 99.18% w/w log Pow 2.7 at 40 °C pH not reported	[Turner 2012c, report no. JSM0267]	OECD 117; HPLC Estimation Method at 40 °C (HPLC column oven temperature). Reference compounds were analysed under the same conditions.
Octanol/water partition coefficient:	purity: 99.18% w/w $logP_{ow}$ = 2.8 in pH 5 buffer (0.2 M potassium dihydrogen orthophosphate-0.07 M disodium hydrogen orthophosphate) $logP_{ow}$ = 2.4 in pH 7 buffer (0.2 M potassium dihydrogen orthophosphate-1.0 M sodium hydroxide) $logP_{ow}$ = 2.0 in pH 9 buffer (0.1 M boric acid in 0.1 M potassium chloride-1.0 M sodium hydroxide)	[Turner 2013a, report no. JSM0505]	OECD 117; HPLC Estimation Method
Water solubility:	purity: 99.45% w/w 0.15 mg/L in water at 20 °C	[Turner 2011, report no. JSM0069]	OECD 105; Column Elution Method
Water solubility	purity: 99.18% w/w 0.12 mg/L at 20 °C in pH 5 0.10 mg/L at 20 °C in pH 7 buffer 0.18 mg/L at 20 °C in pH 9 buffer	[Turner 2013b, report no. JSM0504]	OECD 105; Column Elution Method
Solubility in organic solvents:	purity: 99.18% w/w n-Heptane: 0.00011 g/L Xylene: 0.17 g/L 1,2-Dichloroethane: 4.4 g/L Acetone: 11 g/L Methanol: 4.5 g/L n-Octanol: 1.4 g/L Ethyl acetate: 3.6 g/L	[Turner 2012d, report no. JSM0231]	US EPA OPPTS 830.7840 based on OECD 105; Flask Method at 20 °C
Specific gravity:	purity 99.18% w/w $D^{20}_{4=}$ 1.60 (relative density)	[Turner 2012a, report no. JSM0235]	OECD 109; Determined relative to water using pycnometer
Hydrolysis:	purity: 99.18% w/w Stable at pH 4, 7 and 9 at 50 °C (5 days)	[Unsworth H, 2010, report no. JSM0051]	OECD 111; 0.35 mg/L sterile buffered solutions of ^{14}C-phenyl- or ^{14}C-pyridyl labelled cyclaniliprole (purity > 97%) were kept in the dark (0-5 days for pH 4, 7, and 9).
Photolysis:	no study needed (FAO manula Table 3.3) for foliar applications		
Dissociation constant:	purity: 99.18% w/w pKa = 8.6 at 20 °C	[Turner 2013c, report no. JSM0236]	OECD 112; Spectrophotometric Method
Decomposition or sublimation	purity: 99.18% w/w no endothermic/exothermic reactions below 150 °C. Thermally stable at room temperature	[Turner 2012e, report no. JSM0242]	OECD 113; Differential Scanning Calorimetry

Remark of the reviewer: It is noted that the hydrolysis products found in the processing studies were not taken into account as standards.

Technical material (cyclaniliprole)

Parameter	Result	References	Guidelines
Appearance:	purity 95.71% w/w odourless white powder containing friable agglomerates at 20 °C	[Turner B, 2012f, report no. JSM0228]	US EPA OPPTS 830.6302 and 830.6303 visual, Munsell colour system (N9.25/84.2% R)
Relative density:	purity 95.71% w/w $D^{20}_{4} = 1.57$	[Turner B, 2013d, report no. JSM0230]	US EPA OPPTS 830.7300; Determined relative to water using pycnometer
Solubility in organic solvents:	purity: 95.71% w/w n-Heptane: 0.0001 g/L Xylene: 0.20 g/L 1,2-Dichloroethane: 4.4 g/L Acetone: 10 g/L Methanol: 4.0 g/L n-Octanol: 1.5 g/L Ethyl acetate: 3.6 g/L	[Turner B, 2012g, report no. JSM0232]	US EPA OPPTS 830.7840 based on OECD 105; Flask Method at 20 °C
Boiling point:	purity: 99.18% w/w decomposition soon after melting	[Turner B., 2012a, report no. JSM0235]	OECD 103; Siwoloboff Method

Formulations

Cyclaniliprole has not been evaluated by JMPS and therefore no FAO specifications for technical and formulated cyclaniliprole have been published.

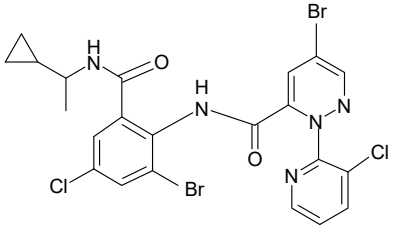
Formulations authorised for use:

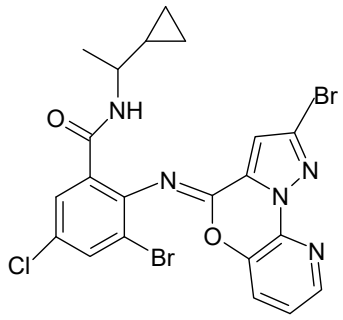
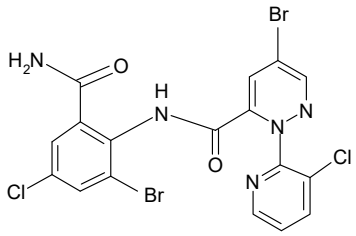
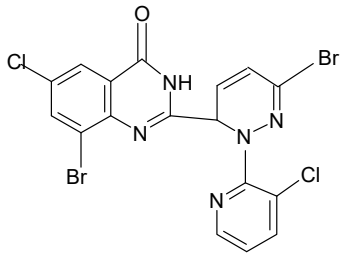
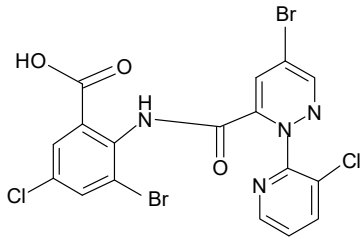
Country	Product name	active ingredient	Submission date/approval date
Korea	Lapitan	4.5% cyclaniliprole, SL formulation	Submission date: 17/09/2015 Approval date: 06/01/2017
USA	CYCLANILIPROLE 50 SL INSECTICIDE	4.55% cylcaniliprole	Approval date: 03/08/2017 (EPA registration number 71512-26)

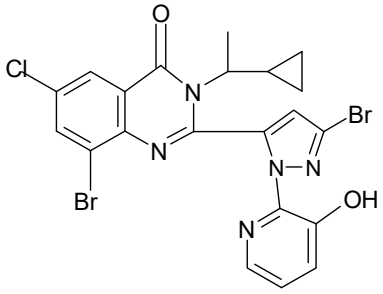
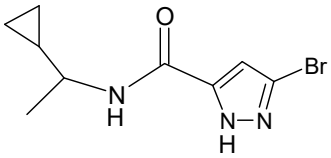
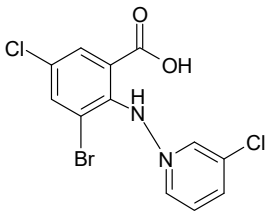
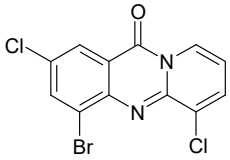
Residue trials have been performed with formulation IKI-3106 50 SL (50 g/L = 4.55% w/w active substance) is a soluble concentrate, a.k.a. IBE 4064.

Conversion factors that can be applied if residues need to be expressed in parent equivalents are based on the respective molecular weights and given in the Table 1.

Table 1 List of reference compounds used in various study reports

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
parent MW=602.11 CF = not applicable	cyclaniliprole IKI-3106 molecular formula: $C_{21}H_{17}Br_2Cl_2N_5O_2$ 3-bromo-*N*-[2-bromo-4-chloro-6-[[(1-cyclopropylethyl)amino]carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide IUPAC: 2',3-dibromo-4'-chloro-1-(3-chloro-2-pyridyl)-6'-{[(1*RS*)-1-cyclopropylethyl]carbamoyl}pyrazole-5-carboxanilide 	goat (all matrices), hen (all matrices) all plant commodities (metabolism studies, field trials, rotational crops studies)

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
NK-1375 MW=565.64 CF = 1.064	molecular formula: $C_{21}H_{16}Br_2ClN_5O_2$ 3-bromo-2-((2-bromo-4*H*- pyrazolo[1,5-d]pyrido[3,2-b]-[1,4]oxazin-4-ylidene)amino)-5-chloro-*N*-(1-cyclopropylethyl)benzamide 	all plant commodities (metabolism studies, field trials if dosed high enough, rotational crops studies) animal metabolism studies: in rat fat
YT-1284 MW=533.99 CF = 1.128	molecular formula: $C_{16}H_9Br_2Cl_2N_5O_2$ 3-bromo-*N*-(2-bromo-6-carbamoyl-4-chlorophenyl)-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-carboxamide 	rat metabolism: bile, urine, liver and kidney animal metabolism: goat (milk, liver, kidney, muscle, fat); chicken (egg, fat, skin, muscle, liver) plant metabolism: apple, lettuce. Not found in field residue studies or rotational crop studies.
NSY-28 MW=516.98 CF = 1.165	molecular formula: $C_{16}H_8Br_2Cl_2N_5O$ 8-bromo-2-(3-bromo-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-yl)-6-chloroquinazolin-4(3*H*)-one 	rat metabolism: bile, urine, plasma, liver, kidney and fat goat (milk, liver, kidney, muscle, fat) chicken (egg, fat, skin, muscle, liver)
NSY-27 MW=534.97 CF = 1.125	molecular formula: $C_{16}H_8Br_2Cl_2N_5O$ 3-bromo-2-(3-bromo-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-carboxamido)-5-chlorobenzoic acid 	rat metabolism: bile, urine, plasma and liver goat (milk, liver, kidney, muscle, fat) chicken (egg, fat, skin, muscle, liver)

Abbreviation	Trivial and systematic chemical names Other abbreviations used in study reports	Found as or in
NSY-137 MW=565.64 CF = 1.064	molecular formula: $C_{21}H_{16}Br_2ClN_5O_2$ 8-bromo-2-(3-bromo-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-yl)-6-chloro-3-(1-cyclopropylethyl)quinazolin-4(3*H*)-one 	Used as reference substance in animal and plant (apple, lettuce and, potato) metabolism studies. Only detected in hen liver tissue.
YT-1327	3-bromo-*N*-(1-cyclopropylethyl)-1*H*-pyrazole-5-carboxamide 	hydrolysis study Processed commodities: tomato paste. Not found in processed apples, peaches and wine Not found in other plant or animal commodity studies.
BCPBA	3-bromo-5-chloro-2-((3-chloropyridin-2-yl)amino)benzoic acid 	hydrolysis study Processed commodities: tomato paste. Not found in processed apples, peaches and wine Not found in other plant or animal commodity studies.
BPQO	4-bromo-2,6-dichloro-11*H*-pyrido[2,1-*b*]quinazolin-11-one 	hydrolysis study Processed commodities: tomato paste. Not found in processed apples, peaches and wine Not found in other plant or animal commodity studies.

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received information on the fate of cyclaniliprole in livestock, plant commodities, soil and rotational crops. The test items used in the studies were cyclaniliprole ^{14}C uniformly labelled in the phenyl ring or at the pyrazole ring as shown in Figure 1.

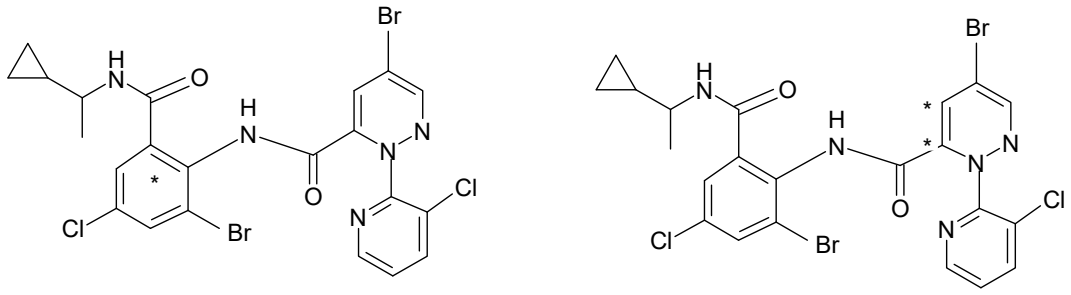


Figure 1 Position of the [^{14}C]-radiolabel in cyclaniliprole; * denotes the position of the radiolabel.

Plant metabolism

The meeting received plant metabolism studies for cyclaniliprole after spray application on fruits and fruiting vegetables (apple), leafy vegetables (lettuce), and root and tuber vegetables (potato). Cyclaniliprole was applied using ^{14}C-phenyl labelled and ^{14}C-pyrazol labelled cyclaniliprole.

Fruits and fruiting vegetables, metabolism study with apple

Apple trees (variety: Granny Smith), growing under commercial conditions (California, USA), were sprayed three times at a target dose rate of 100 g ai/ha [Crowe, 2013a, report JSM0053]. Two radiolabelled forms were used in the study: ^{14}C-phenyl-cyclaniliprole and ^{14}C-pyrazol-cyclaniliprole. The applications were conducted at BBCH growth stage 74 (100 days before final harvest), BBCH 77 (72 days before final harvest) and BBCH 79 (30 days before final harvest).The achieved application rates ranged from 95.5 to 99.5 g ai/ha for the [^{14}C-Ph]-cyclaniliprole and from 91 to 94 g ai/ha for the [^{14}C-Pz]-cyclaniliprole. Fruit and leaves were sampled at an early (immature) stage (BBCH 81, 15 days after the last application (DALA)), and at normal harvest (BBCH 89, 30 days after the last application). In addition, selected fruits were protected from exposure to the spray to study the extent of translocation, which were sampled at normal harvest.

The initial analysis of samples was carried out within four months of sampling. Further analysis of selected sample extracts (the leaf wash and leaf extract from the 15 DALA plants treated with [^{14}C-phenyl-cyclaniliprole) was carried out up to four months later. There were no differences in the metabolite profiles, indicating that no degradation occurs between initial and final analysis under frozen conditions (≤-18 °C).

Samples were surface washed twice with acetonitrile on the day of sampling, after which the two washes were combined. Subsequently, fruit was separated into peel and flesh, and all samples were homogenised and then radio-assayed by LSC. Total radioactive residues (TRR) were determined by summing up the radioactivity recovered in the washes and homogenised plants. The TRR is shown in Table 2 and Table 3. The TRR was higher in leaves compared to fruit, while no residues were detected in protected fruit, indicating that there was no translocation of radioactivity. Both radiolabels gave similar results. The concentrations in apple flesh are below the trigger value of 0.01 mg/kg eq and as such no further work was conducted on apple fruit.

Homogenised samples were extracted with acetonitrile (extracts 1 and 2), acetonitrile : water (1:1 v/v) (extracts 3 and 4), and acetonitrile : water (1:4 v/v) (extract 5). Extracts were combined and after concentration, analysed by HPLC and TLC. Surface washes were also analysed by HPLC and TLC. Reference standards used were NSY-27, NSY-28, YT-1284, NK-1375, NSY-137, and NU-221.

The proportions of radioactivity in surface washes and extracts of samples are summarised in Table 2 and Table 3. In all samples the majority of radioactivity was present in surface washes with the majority of the remaining radioactivity extractedable with solvents. The proportion of radioactivity in the surface washes declined from 15 DALA to 30 DALA, while the proportion of

radioactivity in the peel and flesh increased from 15 DALA to 30 DALA. This indicates some transfer of residues from the surface into the fruit.

The identification and characterisation of radioactive residues in surface wash and peel are shown in Table 4 and in apples leaves in Table 3. Most of the radioactive residue was attributable to parent and NK-1375. Low levels of YT-1284 were measured. The remainder was composed of polar material and minor unidentified components. Both radiolabels gave a similar metabolite profile. Unidentified components in leaves were present up to 1.23 mg/kg eq, but considered not relevant since apple leaves are not used for consumption.

Table 2 Total Radioactive Residues and extractability in apple fruit

Radiolabel	Fraction	Immature harvest (15 DALA)		Mature harvest (30 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
^{14}C-phenyl-cyclaniliprole	Surface wash	92.4	0.137	59.4	0.025
	Flesh	2.3	0.003	11.7	0.005
	Extracts	*1.8*	*0.003*	*9.5*	*0.004*
	Post-extraction solid	*0.5*	*< 0.001*	*2.2*	*0.001*
	Peel	5.3	0.008	28.9	0.012
	Extracts	*3.5*	*0.005*	*20.5*	*0.009*
	Post-extraction solid	*1.8*	*0.003*	*8.4*	*0.004*
	Total extracted	97.7		89.4	
	Total radioactive residue	100	0.148	100	0.042
^{14}C-pyrazol-cyclaniliprole	Surface wash	73.7	0.099	64.0	0.023
	Flesh	8.2	0.011	9.2	0.003
	Extracts	*6.9*	*0.009*	*7.6*	*0.002*
	Post-extraction solid	*1.3*	*0.002*	*1.6*	*0.001*
	Peel	18.0	0.024	26.8	0.010
	Extracts	*14.6*	*0.019*	*19.4*	*0.007*
	Post-extraction solid	*3.4*	*0.005*	*7.4*	*0.003*
	Total extracted	95.3		91	
	Total radioactive residue	100	0.135	100	0.036

Table 3 Total Radioactive Residues and extractability in apple leaves

	Fraction	Immature harvest (15 DALA)		Mature harvest (30 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
^{14}C-phenyl-cyclaniliprole	Surface wash	90.7	17.11	67.3	5.50
	Leaves	9.3	1.76	32.7	2.67
	Extracts	*5.6*	*1.06*	*22.8*	*1.86*
	Post-extraction solid	*3.7*	*0.70*	*9.9*	*0.81*
	Total extracted	96.3		90.1	
	Total radioactive residue	100	18.87	100	8.17
^{14}C-pyrazol-cyclaniliprole	Surface wash	72.5	8.12	84.4	4.57
	Leaves	27.5	3.08	15.6	0.85
	Extracts	*20.7*	*2.32*	*7.5*	*0.41*
	Post-extraction solid	*6.8*	*0.76*	*8.1*	*0.44*
		93.2		91.9	
	Total radioactive residue	100	11.21	100	5.42

Table 4 Identification and characterisation of residues in apple fruit

		^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
	Residue	Immature harvest (15 DALA)		Mature harvest (30 DALA)		Immature harvest (15 DALA)		Mature harvest (30 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Surface wash	Cyclaniliprole	49.3	0.073	35.3	0.015	42.5	0.057	34.8	0.013
	NK-1375	27.3	0.040	17.8	0.008	20.9	0.028	21.6	0.008
	YT-1284	3.9	0.006	nd	nd	1.3	0.002	1.0	< 0.001
	Polar material	1.1[a]	0.002	0.5	< 0.001	2.5	0.003	0.4	< 0.001
	A-Met-B (Rt~12 min.)	0.7	0.001	nd	nd	nd	nd	nd	nd

		^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
Surface wash	Residue	Immature harvest (15 DALA)		Mature harvest (30 DALA)		Immature harvest (15 DALA)		Mature harvest (30 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
	A-Met-C (Rt~14 min.)	0.6	0.001	nd	nd	nd	nd	nd	nd
	A-Met-D-a (Rt~20 min.)	4.7	0.007	1.9	0.001	3.3	0.004	2.7	0.001
	A-Met-D-b (Rt~20 min.)	3.4	0.005	1.8	0.001	2.0	0.003	1.7	0.001
	Others	1.3	0.002	2.0	0.001	1.3	0.002	1.7	0.001
	Total analysed surface wash	92.4	0.137	59.4	0.025	73.7	0.099	64.0	0.023
Extracts	Cyclaniliprole	1.1	0.002	7.7	0.003	4.7	0.006	4.8	0.002
	NK-1375	1.3	0.002	5.6	0.002	3.7	0.005	3.1	0.001
	YT-1284	nd	nd	1.2	0.001	0.5	0.001	nd	nd
	Polar material	0.7	0.001	2.1	0.001	2.8	0.004	9.0	0.003
	A-Met-A (Rt~10 min.)	nd	nd	nd	nd	1.0	0.001	nd	nd
	A-Met-C (Rt~14 min.)	nd	nd	nd	nd	0.5	0.001	nd	nd
	A-Met-D-a (Rt~20 min.)	0.2	0.001	1.7	0.001	0.6	0.001	1.2	< 0.001
	A-Met-D-b (Rt~20 min.)	0.2	< 0.001	1.0	< 0.001	0.4	< 0.001	1.3	< 0.001
	A-Met-F (Rt~31 min.)	nd	nd	1.2	0.001	0.3	< 0.001	nd	nd
	Others	nd	nd	nd	nd	0.1	< 0.001	nd	nd
	Total analysed peel extract	3.5	0.005	20.5	0.009	14.6	0.019	19.4	0.007
	Post extraction solids	1.8	0.003	8.4	0.004	3.4	0.005	7.4	0.003
Total fruit (surface wash + peel)	Cyclaniliprole	50.4	0.075	43.0	0.018	47.2	0.063	39.6	0.015
	NK-1375	28.6	0.042	23.4	0.010	24.6	0.033	24.7	0.009
	YT-1284	3.9	0.006	1.2	0.001	1.8	0.003	1.0	< 0.001
	Polar material[1]	1.8	0.003	2.6	0.001	5.3	0.007	9.4	0.003
	A-Met-A (Rt~10 min.)	nd	nd	nd	nd	1.0	0.001	nd	nd
	A-Met-B (Rt~12 min.)	0.7	0.001	nd	nd	nd	nd	nd	nd
	A-Met-C (Rt~14 min.)	0.6	0.001	nd	nd	0.5	0.001	nd	nd
	A-Met-D-a (Rt~20 min.)	4.9	0.008	3.6	0.002	3.9	0.005	3.9	0.001
	A-Met-D-b (Rt~20 min.)	3.6	0.005	2.8	0.001	2.4	0.003	3.0	0.001
	A-Met-F (Rt~31 min.)	nd	nd	1.2	0.001	0.3	< 0.001	nd	nd
	Others	1.3	0.002	2.0	0.001	1.4	0.002	1.7	0.001
	Total analysed	95.9	0.142	79.9	0.033	88.3	0.118	83.4	0.030
	Un-analysed extracts (flesh)	1.8	0.003	9.5	0.004	6.9	0.009	7.6	0.002
	Total unextracted residue[b]	2.3	0.003	10.6	0.005	4.7	0.007	9.0	0.004

nd: not detected; Rt: HPLC retention time; Others: Radioactivity distributed throughout regions of the chromatogram other than those specified and which did not contain any discrete radioactive components

[a] Shown to comprise of up to five separate components

[b] Total un-extracted residue consists of un-extracted residue from peel and un-extracted residue from flesh (see Table 2)

Table 5 Identification and characterisation of residues in apple leaves

		^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
Fraction	Residue	Immature harvest (15 DALA)		Mature harvest (30 DALA)		Immature harvest (15 DALA)		Mature harvest (30 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Surface wash	Cyclaniliprole	50.2	9.46	49.1	4.02	40.2	4.50	48.2	2.61
	NK-1375	23.8	4.48	7.5	0.62	21.3	2.39	22.2	1.20
	YT-1284	1.2	0.22	0.7	0.06	1.3	0.15	1.0	0.06
	Polar material	0.8	0.15	nd	nd	0.6	0.07	nd	nd
	A-Met-A (Rt~10 min.)	0.6	0.12	nd	nd	nd	nd	nd	nd
	A-Met-B (Rt~12 min.)	0.5	0.09	nd	nd	nd	nd	0.3	0.01
	A-Met-C (Rt~14 min.)	0.8	0.15	0.5	0.04	0.9	0.10	0.6	0.03
	A-Met-D-a (Rt~20 min.)	6.1	1.15	4.2	0.34	4.2	0.47	4.5	0.24
	A-Met-D-b (Rt~20 min.)	3.6	0.68	3.3	0.28	2.2	0.25	3.4	0.19
	A-Met-F (Rt~31 min.)	2.4	0.46	1.1	0.09	nd	nd	2.4	0.13

		^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
	Others	0.7	0.14	0.7	0.06	1.9	0.21	1.8	0.10
	Total analysed surface wash	90.7	17.11	67.3	5.50	72.5	8.12	84.4	4.57
Extracts	Cyclaniliprole	1.0	0.19	8.4	0.69	3.7	0.42	1.5	0.08
	NK-1375	0.3	0.06	6.3	0.52	3.3	0.37	0.8	0.04
	YT-1284	0.8	0.15	1.0	0.09	1.1	0.13	nd	nd
	Polar material	1.2	0.22	1.2	0.10	7.0a	0.79a	5.3	0.29
	A-Met-A (Rt~10 min.)	0.5	0.09	0.5	0.04	1.4	0.16	nd	nd
	A-Met-B (Rt~12 min.)	0.5	0.09	0.7	0.05	0.9	0.10	nd	nd
	A-Met-C (Rt~14 min.)	0.5	0.09	0.9	0.07	1.1	0.13	nd	nd
	A-Met-D-a (Rt~20 min.)	0.4	0.08	1.6	0.13	1.1	0.12	nd	nd
	A-Met-D-b (Rt~20 min.)	0.3	0.06	1.0	0.08	0.6	0.07	nd	nd
	A-Met-F (Rt~31 min.)	0.2	0.04	1.0	0.08	0.5	0.05	nd	nd
	Others	nd	nd	0.2	0.02	nd	nd	nd	nd
	Total analysed leaf extracts	5.6	1.06	22.8	1.86	20.7	2.32	7.5	0.41
	Un-extracted residue	3.7	0.70	9.9	0.81	6.8	0.76	8.1	0.44
Total leaves (surface wash + extract)	Cyclaniliprole	51.2	9.65	57.5	4.71	43.9	4.92	49.7	2.69
	NK-1375	24.1	4.54	13.8	1.14	24.6	2.76	23.0	1.24
	YT-1284	2.0	0.37	1.7	0.15	2.4	0.28	1.0	0.06
	Polar material	2.0[1]	0.37	1.2[1]	0.10	7.6	0.86	5.3	0.29
	A-Met-A (Rt~10 min.)	1.1	0.21	0.5	0.04	1.4	0.16	nd	nd
	A-Met-B (Rt~12 min.)	1.0	0.18	0.7	0.05	0.9	0.10	0.3	0.01
	A-Met-C (Rt~14 min.)	1.3	0.24	1.4	0.11	2.0	0.23	0.6	0.03
	A-Met-D-a (Rt~20 min.)	6.5	1.23	5.8	0.47	5.3	0.59	4.5	0.24
	A-Met-D-b (Rt~20 min.)	3.9	0.74	4.3	0.36	2.8	0.32	3.4	0.19
	A-Met-F (Rt~31 min.)	2.6	0.50	2.1	0.17	0.5	0.05	2.4	0.13
	Others	0.7	0.14	0.9	0.08	1.9	0.21	1.8	0.10
	Total analysed	96.3	18.17	90.1	7.36	93.2	10.44	91.9	4.98
	Un-extracted residue	3.7	0.70	9.9	0.81	6.8	0.76	8.1	0.44

nd: not detected; Rt: HPLC retention time; Others: Radioactivity distributed throughout regions of the chromatogram other than those specified and which did not contain any discrete radioactive components

Leafy vegetables, metabolism study with lettuce

Lettuce (variety: Little Gem), sown in pots and grown to maturity in plastic tunnels, was sprayed three times at a target dose rate of 100 g ai/ha [Crowe, 2013b, report JSM0054]. Two radiolabelled forms were used in the study: ^{14}C-phenyl-cyclaniliprole and ^{14}C-pyrazol-cyclaniliprole. The applications were conducted 35 (BBCH 13), 25 (BBCH 16) and 15 days (BBCH 41) before harvest. The achieved application rates were in the range 107.3 to 115.9 g ai/ha for the [^{14}C-Ph]-cyclaniliprole and 107.5 to 117.1 g ai/ha for the [^{14}C-Pz]-cyclaniliprole. Lettuces were sampled at immature and mature harvest, 8 (BBCH 46) and 15 days (BBCH 49) after the last application (8 and 15 DALA). Whole lettuce plants were sampled by cutting just above the surface of the soil.

The initial analysis of samples was carried out within three months of sampling. Further analysis of selected sample extracts (surface washes and lettuce extracts) was carried out about two months later. There were no differences in the metabolite profiles, indicating that the samples were stable under frozen conditions (≤-18 °C) from initial analysis until final analysis.

Lettuces were surface washed twice with acetonitrile on the day of sampling, after which the two washes were combined. Lettuce samples were homogenised and combusted. The sum of the radioactivity recovered in the washes and the homogenised plant material gave the total radioactive residue (TRR) in each sample. The TRR is shown in Table 6. The TRR in the lettuces declined from the immature harvest to the mature harvest as the plants developed. Both radiolabels gave similar results.

The samples were then extracted by homogenisation with acetonitrile and subsequently acetonitrile:water (1:1 v/v). Extracts were combined are analysed by LSC and HPLC. Reference

standards used were NSY-27, NSY-28, YT-1284, NK-1375, and NSY-137.The proportions of radioactivity in surface washes and extracts of samples are summarised in Table 7. Following surface washing, most of the remaining radioactive residues could be extracted. The identification and characterisation of radioactive residues are shown in Table 7. Most of the radioactive residue was attributable to cyclaniliprole and NK-1375. The levels and patterns of metabolites were similar in the two treatment groups, except that application of ^{14}C-pyrazol-cyclaniliprole resulted in the production of more polar metabolites.

Table 6 Total Radioactive Residues and extractability in lettuce

Radiolabel	Fraction	8 DALA		15 DALA	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
^{14}C-phenyl-cyclaniliprole	Surface wash	84.3	0.637	76.4	0.300
	Acetonitrile extract	9.8	0.074	11.2	0.044
	Acetonitrile : water extract	3.2	0.024	6.3	0.025
	Post-extraction solid	2.7	0.020	6.1	0.024
	Total radioactive residue	100	0.756	100	0.393
^{14}C-pyrazol-cyclaniliprole	Surface wash	83.4	0.638	77.3	0.287
	Acetonitrile extract	7.8	0.060	12.2	0.045
	Acetonitrile : water extract	5.9	0.045	5.2	0.019
	Post-extraction solid	2.9	0.023	5.3	0.020
	Total radioactive residue	100	0.765	100	0.371

Table 7 Identification and characterisation of residues in lettuce

		^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
Fraction	Residue	8 DALA		15 DALA		8 DALA		15 DALA	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Surface wash	Cyclaniliprole	68.9	0.520	54.3	0.213	65.1	0.498	50.6	0.188
	NK-1375	10.9	0.082	14.6	0.057	12.8	0.098	18.5	0.069
	Metabolite E (Rt~28 min)	nd	nd	1.2	0.005	nd	nd	1.1	0.004
	Others	4.6	0.034	6.3	0.025	5.5	0.042	7.2	0.027
	Total analysed in surface wash	84.4	0.636	76.4	0.3	83.4	0.638	77.4	0.288
Acetonitrile:water extracts	Cyclaniliprole	8.8	0.067	10.4	0.041	8.9	0.068	8.8	0.032
	NK-1375	1.8	0.014	3.0	0.012	2.0	0.015	3.3	0.012
	YT-1284	0.4	0.003	0.6	0.003	0.3	0.002	0.6	0.002
	Polar material	0.3	0.002	0.5	0.002	0.3	0.003	0.7	0.002
	Metabolite A (Rt~7 min)	nd	nd	nd	nd	0.2	0.002	0.5	0.002
	Metabolite B (Rt~9 min)	nd	nd	nd	nd	0.3	0.003	0.6	0.002
	Metabolite C (Rt~15 min)	nd	nd	nd	nd	0.3	0.002	0.5	0.002
	Metabolite D (Rt~20 min)	0.8	0.006	1.2	0.005	0.6	0.005	1.2	0.004
	Metabolite E (Rt~28 min)	0.5	0.004	1.0	0.004	nd	nd	nd	nd
	Others	0.4	0.003	0.8	0.003	0.8	0.006	1.1	0.004
	Total analysed in extracts	13	0.099	17.5	0.07	13.7	0.106	17.3	0.062
Total plant (surface wash + extracts)	Cyclaniliprole	77.7	0.587	64.7	0.254	74.0	0.566	59.4	0.220
	NK-1375	12.7	0.096	17.6	0.069	14.8	0.113	21.8	0.081
	YT-1284	0.4	0.003	0.6	0.003	0.3	0.002	0.6	0.002
	Polar material	0.3	0.002	0.5	0.002	0.3	0.003	0.7	0.002
	Metabolite A (Rt~7 min)	nd	nd	nd	nd	0.2	0.002	0.5	0.002
	Metabolite B (Rt~9 min)	nd	nd	nd	nd	0.3	0.003	0.6	0.002
	Metabolite C (Rt~15 min)	nd	nd	nd	nd	0.3	0.002	0.5	0.002
	Metabolite D (Rt~20 min)	0.8	0.006	1.2	0.005	0.6	0.005	1.2	0.004
	Metabolite E (R ~28 min)	0.5	0.004	2.2	0.009	nd	nd	1.1	0.004
	Total analysed	97.4	0.735	93.9	0.37	97.1	0.744	94.7	0.350
	PES	2.7	0.020	6.1	0.024	2.9	0.023	5.3	0.020
	Total [a]	100.1	0.755	100	0.394	100	0.767	100	0.370

nd: not detected; Rt: HPLC retention time; PES = post-extraction solids.

Others: Radioactivity distributed throughout regions of the chromatogram other than those specified and which did not contain any discrete radioactive components

[a] Totals calculated by reviewer with rounding leading to slight deviations.

Root and tuber vegetables, metabolism study with potato

Seed potatoes (variety: *Estima Second Early*) were sown in sandy loam soil pots and grown to maturity under netting in the UK (Warwickshire) in 2010 and treated with formulation of cyclaniliprole to investigate the metabolism of cyclaniliprole [Crowe, 2013c, report JSM0055]. Two radiolabelled forms were used in the study: ^{14}C-phenyl-cyclaniliprole and ^{14}C-pyrazol-cyclaniliprole. Plants were treated three times at a field application rate of 40 g ai/ha per application, or 4 mg/m^2 assuming a crop density of 8 plants per square metre. The applications were conducted at BBCH growth stage 46 (43 days before final harvest), BBCH 91 (29 days before final harvest) and BBCH 93 (15 days before final harvest). Whole plants (4 per group) were samples at early (immature) stage (BBCH 96, 8 days after last application) and at normal harvest (BBCH 99, 15 days after the last application). Foliage was sampled by cutting all of the material above the surface of the soil. Tubers were removed from the soil and remaining particles were washed off the surface of the tuber with water. Tubers and foliage from each group were divided into three batches and each batch weighed.

The initial analysis of samples was carried out within three months of sampling. Further HPLC analysis of selected sample extracts was carried out to two months later. The HPLC chromatograms showed no evidence of degradation after two months storage, indicating that the samples were stable under frozen conditions (≤-18 °C) from initial to final analysis.

Samples of foliage were surface washed twice with acetonitrile on the day of sampling, after which the two washes were combined. Tubers were broken up before homogenising with solid carbon dioxide. The foliage samples were homogenised with solid carbon dioxide as well. After evaporation of the carbon dioxide the homogenized samples were weighed. And replicate samples were removed for combustion to determine the radioactivity content by LSC.

Total radioactive residues (TRR) were determined by summing up the radioactivity recovered in the washes and homogenised foliage samples. For the tubers the radioactivity recovered in the homogenised samples provided the total residue. The TRR is shown in Table 8 and Table 9. The TRR was higher in leaves (1.801 (mature)-2.359 (immature) mg/kg eq with ^{14}C-Ph and 1.574 (mature)-3.023 (immature) mg/kg eq with ^{14}C-Pz) compared to tubers (0.001 and 0.002 mg/kg eq ^{14}C-Ph and ^{14}C-Pz, respectively). The concentrations in tubers are below the trigger value of 0.01 mg/kg eq and as such no further work was conducted on tubers.

Homogenised samples of foliage were extracted twice with either acetonitrile (extracts 1 and 2), acetonitrile:water (1:1 v/v) (extracts 3 and 4), or acetonitrile:water (1:4 v/v) (extract 5). Radioactivity content of the combined extracts was determined by LSC after each extraction. Post extraction solids were air dried, homogenised, weighed and five replicate aliquots were removed for combustion to determine radioactivity content by LSC. Extracts and surface washes were also analysed by HPLC and TLC for identification and characterisation. Reference standards used were NSY-27, NSY-28, YT-1284, NK-1375, and NSY-137.

The proportions of radioactivity in surface washes and extracts of samples are summarised in Table 8 and Table 9. In all samples the majority of radioactivity was present in surface washes with the majority of the remaining radioactivity extractedable with solvents. The proportion of radioactivity in the surface washes and foliage did not change significantly. This may indicate that there is no transfer of residues from the surface into the tuber within a time frame of 1 week.

The identification and characterisation of radioactive residues are shown in Table 10. Most of the radioactive residue was attributable to parent and NK-1375. Low levels of YT-1284 were measured. The remainder was composed of polar material and minor unidentified components. Both radiolabels gave a similar metabolite profile.

Table 8 Total Radioactive Residues and extractability in potato foliage

Radiolabel	Fraction	Immature harvest (8 DALA)		Mature harvest (15 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
^{14}C-phenyl-cyclaniliprole	Surface wash	54.1	1.275	52.7	0.949
	homogenized foliage	39			
	acetonitrile	30.6	0.721	29.3	0.527
	acetonitrile:water (1:1 v:v)	7.3	0.173	7.2	0.130
	acetonitrile:water (1:4 v/v)	1.1	0.027	1.1	0.019
	Post-extraction solid	6.9	0.163	9.7	0.176
	Total radioactive residue	100	2.359	100	1.801
^{14}C-pyrazol-cyclaniliprole	Surface wash	57.0	1.722	43.6	0.686
	homogenized foliage	37.2			
	acetonitrile	30.8	0.930	38.6	0.608
	acetonitrile:water (1:1 v:v)	5.6	0.170	8.5	0.134
	acetonitrile:water (1:4 v/v)	0.8	0.023	1.1	0.018
	Post-extraction solid	5.9	0.178	8.2	0.128
	Total radioactive residue	100	3.023	100	1.574

Table 9 Identification and characterisation of residues in potato foliage

		^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
Fraction	Residue	Immature harvest (8 DALA)		Mature harvest (15 DALA)		Immature harvest (8DALA)		Mature harvest (15 DALA)	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Surface wash	*Total*[a]	*54.2*	*1.276*	*52.6*	*0.949*	*57.1*	*1.722*	*43.5*	*0.686*
	Cyclaniliprole	41.6	0.981	37.9	0.683	41.5	1.254	33.7	0.531
	NK-1375	6.9	0.162	7.3	0.132	8.6	0.258	6.1	0.096
	Metabolite F (Rt~18 min.)	1.0	0.023	1.6	0.028	1.4	0.041	nd	nd
	Metabolite E (Rt~28 min.)	1.0	0.023	1.2	0.022	1.2	0.036	0.7	0.012
	Others	3.7	0.087	4.6	0.084	4.4	0.133	3.0	0.047
Foliage	*Extracts [a]*	*39*	*0.921*	*37.7*	*0.677*	*37.3*	*1.124*	*48.2*	*0.76*
	Cyclaniliprole	25.7	0.607	22.2	0.400	23.1	0.699	29.5	0.465
	NK-1375	6.2	0.147	6.8	0.122	6.4	0.194	8.4	0.132
	Polar material	0.5	0.013	0.9	0.016	0.6	0.017	1.4	0.022
	Metabolite A (Rt~15 min.)	nd	nd	nd	nd	0.6	0.017	0.8	0.013
	Metabolite C (Rt~15 min.)	0.4	0.009	0.8	0.014	0.6	0.017	0.8	0.012
	Metabolite F (Rt~18 min.)	2.0	0.046	1.9	0.034	1.6	0.047	1.5	0.024
	Metabolite D (Rt~20 min.)	1.7	0.041	1.7	0.030	1.8	0.055	1.9	0.030
	Metabolite E (Rt~28 min.)	2.1	0.050	2.7	0.049	1.4	0.042	2.4	0.038
	Others	0.4	0.008	0.7	0.012	1.2	0.036	1.5	0.024

nd: not detected; Rt: HPLC retention time

[a] Totals calculated

Table 10 Identification and characterisation of residues in potato leaves (surface wash and extract combined)

	^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
Residue	Immature harvest (8 DALA)		Mature harvest (15 DALA)		Immature harvest (8 DALA)		Mature harvest (15 DALA)	
	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Cyclaniliprole	67.3	1.588	60.1	1.083	64.6	1.953	63.2	0.996
NK-1375	13.1	0.309	14.1	0.254	15	0.452	14.5	0.228
Polar material	0.5	0.013	0.9	0.016	0.6	0.017	1.4	0.022
Metabolite A (Rt~15 min.)	nd	nd	nd	nd	0.6	0.017	0.8	0.013
Metabolite C (Rt~15 min.)	0.4	0.009	0.8	0.014	0.6	0.017	0.8	0.012
Metabolite F (Rt~18 min.)	3	0.069	3.5	0.062	3	0.088		

Residue	^{14}C-phenyl-cyclaniliprole				^{14}C-pyrazol-cyclaniliprole			
	Immature harvest (8 DALA)		Mature harvest (15 DALA)		Immature harvest (8 DALA)		Mature harvest (15 DALA)	
	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Metabolite D (Rt~20 min.)	1.7	0.041	1.7	0.03	1.8	0.055	1.9	0.03
Metabolite E (Rt~28 min.)	3.1	0.073	3.9	0.071	2.6	0.078	3.1	0.05
Others	4.1	0.095	5.3	0.096	5.6	0.169	4.5	0.071
Total analysed[a]	93.2	2.197	90.3	1.626	94.4	2.846	91.7	1.446
Un-extracted residue	6.9	0.163	9.7	0.176	5.9	0.178	8.2	0.128
Total[a]	100.1	2.36	100	1.802	100.3	3.024	99.9	1.574

nd: not detected; Rt: HPLC retention time
[a] Values calculated from Tables 8 and 9

Overview of the metabolic pathway in plants

Metabolism studies conducted with crops representative of fruits and fruiting vegetables (apple), leafy vegetables (lettuce) and root and tuber vegetables (potatoes) and based on foliar treatment, show that the metabolic pathways in the three crop groups are similar and simple via two main pathways;

- Cyclization by reaction between an oxygen moiety and a chloride moiety, giving the compound NK-1375 (pathway 1, major pathway)
- N-de-alkylation of cyclaniliprole; loss of 1-cyclopropylethyl group on the nitrogen atom in amide moiety in the side chain yielding the corresponding primary amide YT-1284 (pathway 2, minor pathway).

In apple, lettuce, potato the major compound in the residue is parent (40–78%TRR), followed by the metabolite NK-1375 (13–29 %TRR)) and finally YT-1284 (0.3–3.9%TRR), the latter not being detected in potato. The proposed biotransformation pathway for cyclaniliprole in plants is shown in Figure 2.

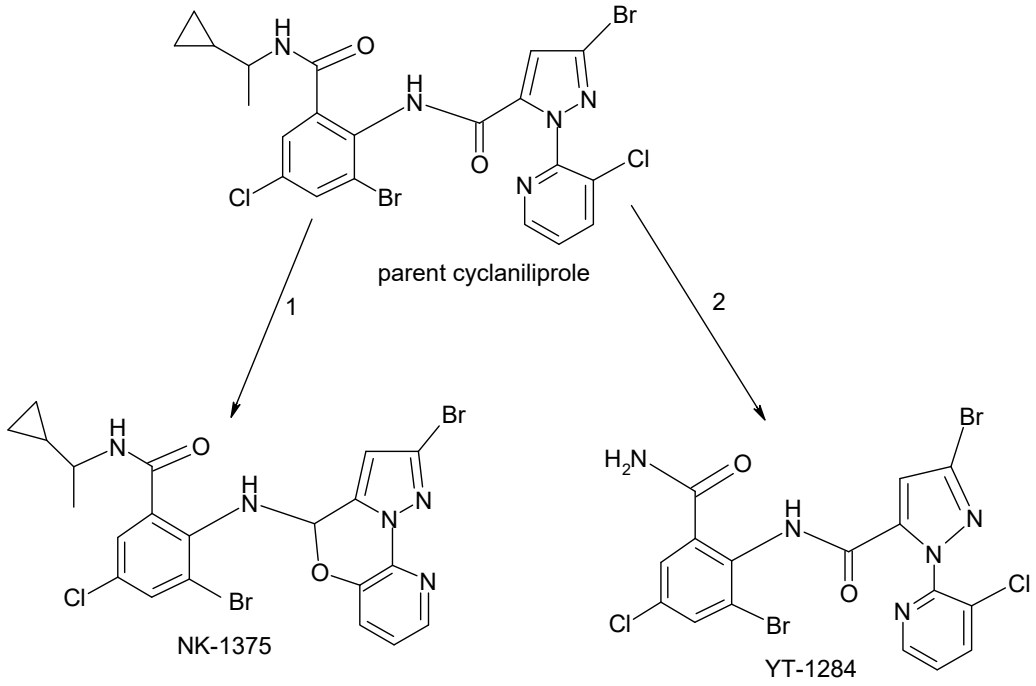


Figure 2 Metabolic pathway of cyclaniliprole in plants

Rotational crops

The Meeting received information on confined and field rotational crops.

Confined rotational crop studies

A confined rotational crop study was designed to provide information on the uptake and metabolism of cyclaniliprole in rotational crops [Crowe, 2013d, report JSM0148]. Cyclaniliprole was applied as ^{14}C-phenyl-cyclaniliprole to soil at a nominal rate equivalent to 100 g ai/ha (the actual application rates are described in Table 12 and ranged from 98–110 g ai/ha). The soil characteristics were: sandy loam, pH 5.2, 3.1% om, 1.8% oc. After ageing the soil for 30, 120 and 365 days, wheat (variety: Tybalt), carrot (variety: Early Nantes 2) and lettuce (variety: Little Gem) were sown and grown to maturity. Samples of soil were taken at the end of each ageing period and at the time of harvest of the mature crops. Crops were sampled and analysed at early harvest and final harvest, with additional wheat forage samples taken as soon as sufficient crop was available. The lettuce (3–4 weeks before normal harvest and at normal harvest), wheat forage (7–8 weeks after sowing) and hay (BBCH 77–83) samples were analysed as whole samples. The final harvest wheat samples (BBCH 89–92) were separated into grain and straw (including chaff) and the carrots (3 weeks before normal harvest and at normal harvest) were separated into foliage and root.

The initial analyses of samples were carried out within about six months of sampling. A representative sample (the extract of hay from wheat grown in soil aged for 120 days) was reanalysed after an interval of approximately four months to confirm stability during storage. There was essentially no difference in the HPLC profile between the initial analysis and the repeat analysis.

The total radioactive residue (TRR) in soil was determined by LSC of aliquots of homogenised soil. As the TRRs were all greater than 0.01 mg/kg eq, samples were extracted with acetonitrile and then acetonitrile:water (1:1, v/v). The radioactivity content of the extracts was determined by LSC. The extracts were combined and after concentration analysed by HPLC with radio-detection. In soil, most of the TRR was extracted (≥ 92.8%). With one exception, cyclaniliprole accounted for >90% of the TRR in all soils analysed. In addition, metabolite NK-1375 was formed (max. 7.5% TRR, 0.003 mg/kg eq), and metabolite C (Rt~25 minutes; max. 1.6% TRR, 0.001 mg/kg eq). Unidentified other components were maximally 4.7% (0.002 mg/kg eq), which consisted of radioactivity that was distributed throughout regions of the chromatogram other than those specified and which did not contain any discrete radioactive components. Results are shown in Table 11.

The TRR was determined in homogenised plant samples by LSC. Results are shown in Table 12.

Samples where the TRR were greater than 0.01 mg/kg eq were processed further. A portion of the crop sample was extracted sequentially with acetonitrile, acetonitrile:water (1:1, v/v) and acetonitrile:water (1:4, v/v) (two extractions with each solvent). The radioactivity content of the extracts was determined by LSC. Combined extracts were concentrated and analysed by HPLC with radiodetection. As the TRRs in all lettuce and carrot samples were below 0.01 mg/kg eq no further characterisation of these residues was required. The results are shown in Tables 11 and 13.

Table 11 Characterisation of the residue in soil

	Component	Container to be used for wheat		Container to be used for lettuce		Container to be used for carrots	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Soil aged for 30 days	IKI-3106	99.4	0.067	98.5	0.027	98.8	0.046
	NK-1375	nd	nd	nd	nd	nd	nd
	Other extracted	0.6	< 0.0005	1.5	< 0.0005	1.2	0.001
	Total extracted	100.0	0.067	100.0	0.027	100.0	0.047
	Post-extraction solid	nd	nd	nd	nd	nd	nd
	TRR	100	0.067	100	0.027	100	0.047

	Component	Container to be used for wheat		Container to be used for lettuce		Container to be used for carrots	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
	Component	Container in which wheat was grown		Container in which lettuce was grown		Container in which carrots were grown	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Soil in which crops were grown in 30-day aged soil at the times of their harvest	IKI-3106	95.0	0.050	92.5	0.034	96.2	0.041
	NK-1375	0.6	< 0.0005	7.5	0.003	1.2	0.001
	Other extracted	0.4	< 0.0005	nd	nd	0.2	< 0.0005
	Total extracted	96.0	0.051	100.0	0.036	97.6	0.042
	Post-extraction solid	4.0	0.002	nd	nd	2.4	0.001
	TRR	100	0.053	100	0.036	100	0.043
	Component	Container to be used for wheat		Container to be used for lettuce		Container to be used for carrots	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Soil aged for 120 days	IKI-3106	94.8	0.052	93.9	0.040	95.1	0.057
	NK-1375	2.0	0.001	1.0	< 0.0005	2.0	0.001
	Other extracted	0.5	< 0.0005	1.7	0.001	0.6	< 0.0005
	Total extracted	97.3	0.053	96.6	0.041	97.6	0.059
	Post-extraction solid	2.7	0.001	3.4	0.001	2.4	0.001
	TRR	100	0.054	100	0.042	100	0.060
		Container in which wheat was grown		Container in which lettuce was grown		Container in which carrots were grown	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Soil in which crops were grown in 120-day aged soil at the times of their harvest	IKI-3106	94.3	0.067	94.9	0.065	91.5	0.058
	NK-1375	nd	nd	nd	nd	nd	nd
	Metabolite C (Rt ~25 minutes)	1.0	0.001	nd	nd	1.6	0.001
	Other extracted	0.7	< 0.0005	1.5	0.001	4.1	0.003
	Total extracted	95.9	0.068	96.4	0.066	97.1	0.062
	Post-extraction solid	4.1	0.003	3.6	0.002	2.9	0.002
	TRR	100	0.071	100	0.068	100	0.064
	Component	Container to be used for wheat					
		[% TRR]	[mg/kg eq]				
Soil aged for 365 days	IKI-3106	92.9	0.041				
	NK-1375	nd	nd				
	Metabolite C (Rt ~25 minutes)	0.5	< 0.0005				
	Other extracted	1.7	0.001				
	Total extracted	95.0	0.042				
	Post-extraction solid	5.0	0.002				
	TRR	100	0.044				
	Component	Container in which wheat was grown					
		[% TRR]	[mg/kg eq]				
Soil in which wheat was grown in 365-day aged soil at the time of harvest	IKI-3106	87.1	0.035				
	NK-1375	nd	nd				
	Metabolite C (Rt ~25 minutes)	0.9	< 0.0005				
	Other extracted	4.7	0.002				
	Total extracted	92.8	0.037				
	Post-extraction solid	7.2	0.003				
	TRR	100	0.040				

nd = not detected

Table 12 Total Radioactive Residues in plant matrices

Crop	Actual application rate (kg ai/ha)	Days of soil ageing	Days after application	Days after sowing	Matrix	TRR [mg/kg eq]
Wheat	0.098	30	80	50	Forage	0.018
			153	123	Hay (BBCH 77-83)	0.030
			176	146	Grain (BBCH 89-92)	0.001
					Straw (BBCH 89-92	0.058
Lettuce	0.110		100	70	Foliage (immature)	0.001
			120	90	Foliage (mature)	0.001
Carrot	0.107		112	82	Foliage (immature)	0.001
					Root (immature)	0.001
			133	103	Foliage (mature)	0.002
					Root (mature)	0.001
Wheat	0.108	120	174	54	Forage	0.018
			233	113	Hay (BBCH 77-83)	0.028
			262	142	Grain (BBCH 89-92)	nd
					Straw (BBCH 89-92	0.059
Lettuce	0.108		190	70	Foliage (immature)	0.001
			219	99	Foliage (mature)	0.001
Carrot	0.104		212	92	Foliage (immature)	0.001
					Root (immature)	0.001
			233	113	Foliage (mature)	0.001
					Root (mature)	0.001
Wheat	0.110	365	414	49	Forage	0.015
			465	100	Hay (BBCH 77-83)	0.017
			498	133	Grain (BBCH 89-92)	nd
					Straw (BBCH 89-92	0.029

nd = not detected

Table 131 Identification and characterisation of residues in wheat grown in soil aged for 30, 120 and 365 days

	Component	Forage		Hay		Straw	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
Wheat grown in soil aged for 30 days	Cyclaniliprole	66.8	0.012	72.3	0.021	74.9	0.043
	NK-1375	13.8	0.003	1.9	0.001	nd	nd
	Polar material	10.8	0.002	nd	nd	nd	nd
	Metabolite F (Rt~18 minutes)	nd	nd	1.2	< 0.0005	2.0	0.001
	Metabolite D (Rt~20 minutes)	nd	nd	1.4	< 0.0005	2.4	0.001
	Others	1.2	< 0.0005	0.7	< 0.0005	6.0	0.003
	Total organo-soluble	92.7	0.017	77.6	0.023	85.2	0.049
	Water soluble fraction	-	-	9.2	0.003	-	-
	Post-extraction solid	7.3	0.001	13.3	0.004	14.8	0.009
	TRR	99.9	0.018	100	0.030	100.1	0.057
Wheat grown in soil aged for 120 days	Cyclaniliprole	90.0	0.016	80.0	0.022	73.9	0.044
	NK-1375	0.6	< 0.0005	2.3	0.001	2.4	0.001
	Metabolite F (Rt~18 minutes)	nd	nd	1.5	< 0.0005	1.9	0.001
	Metabolite D (Rt~20 minutes)	nd	nd	1.5	< 0.0005	2.2	0.001
	Others	0.5	< 0.0005	0.6	< 0.0005	0.9	0.001
	Total organo-soluble	91.2	0.016	85.8	0.024	81.2	0.048
	Water soluble fraction	nd	nd	nd	nd	nd	nd
	Post-extraction solid	8.8	0.002	14.2	0.004	18.8	0.011
	TRR	99.9	0.018	100.1	0.027	100.1	0.059
Wheat grown in soil aged for 365 days	Cyclaniliprole	81.4	0.013	80.6	0.014	78.4	0.024
	NK-1375	2.7	< 0.0005	3.5	0.001	1.3	< 0.0005
	Metabolite F (Rt~18 minutes)	0.4	< 0.0005	0.4	< 0.0005	2.2	0.001
	Metabolite D (Rt~20 minutes)	4.7	0.001	1.0	< 0.0005	2.4	0.001
	Others	nd	nd	0.9	< 0.0005	1.5	< 0.0005
	Total organo-soluble	89.3	0.014	86.4	0.015	85.9	0.026

	Component	Forage		Hay		Straw	
		[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]	[% TRR]	[mg/kg eq]
	Water soluble fraction	nd	nd	nd	nd	nd	nd
	Post-extraction solid	10.7	0.002	13.6	0.002	14.1	0.004
	TRR	99.9	0.016	100	0.017	99.9	0.030

nd = not detected

Others: Radioactivity distributed throughout regions of the chromatogram other than those specified and which did not contain any discrete radioactive components

Field rotational crop studies

Study 1

The magnitude and decline of residues of cyclaniliprole and its metabolite NK-1375 has been investigated in wheat (whole plant, grain and straw) as rotational crop following cultivation of tomatoes or peppers as primary crop [Bartolomé, 2013, report JSM0414]. Tomatoes or peppers were treated with two foliar applications at a nominal rate of 0.04 kg ai/ha with an interval of 10–11 days. The primary crop was incorporated into the soil 1 day after the last application (DALA). Wheat was drilled into the soil 29–32 DALA (plots 1A and 2A) and 124–154 DALA (plots 1B and 2B). Samples were collected from treated (plot 2) and untreated plots (plot 1) at BBCH 31–33 (whole plant, forage), BBCH 75–83 (whole plant, hay) and BBCH 89 (grain and straw) for wheat drilled 29–32 (plots 1A and 2A) and 124–154 (plots 1B and 2B) DALA. No sampling of soil occurred for residue analysis. According to the applicant the study has been performed under guidelines where application to bare soil and soil analysis was not required.

Three trials have been being conducted (in Hungary, Italy and Spain in 2012). Trial characteristics are shown in Table 14. Wheat samples were analysed for parent cyclaniliprole and NK-1375 using HPLC-MS/MS method JSM0269. The duration of frozen storage (-20 °C) was between 12 and 81 days. No cyclaniliprole or NK-1375 was detected above the LOQ in the samples from untreated plots. Control sample < 0.3 LOQ for each analyte. The concurrent recovery was within 70–120% (n=1) for each analyte in each matrix.

Results are shown in Table 15. The results for forage and hay were reported on as receivd basis (i.e. not calculated bbakc to dry matter content). Cyclaniliprole was found at a maximum concentration equal to the LOQ of 0.01 mg/kg in four samples. These were: in the trial in Italy, in the straw of wheat drilled 29 DALA and in the straw of wheat drilled 124 DALA; and in the trial in Spain, in the straw of wheat drilled 32 DALA and in the whole plant (forage) of wheat drilled 128 DALA. In all other samples and the samples from the trial in Hungary cyclaniliprole was below the LOQ. NK-1375 was not detected in any sample from any trial. In wheat from control (untreated) plots, both cyclaniliprole and NK-1375 were below the LOQ.

Table 14 Experimental conditions for field rotational crop studies

Trial & Location	Actual application; last appl date	Soil type	pH	% om	CEC (meq/100 g)	PBI (DALA)	Rotational crops
JSM0414-01; Székesfehérvár, Hungary 2012	0.039 + 0.042 kg ai/ha (plot 2A); 0.042 + 0.042 kg ai/ha (plot 2B), interval 10 days, to peppers (var. Féherözön) 01 Oct 2012	Sandy clay loam	8.1	2.9	13.8	30, 154	Wheat (var. Magdaléna (plot A) / Lona (plot B))
JSM0414-03; Mulazzano, Italy 2012	0.040 + 0.042 kg ai/ha, interval 11 days, to tomato (var. Heinz H1900)	Loam	5.8	1.5	11.4	29, 124	Wheat (var. PR22R58 (plot A) / Blasco (plot B))

Trial & Location	Actual application; last appl date	Soil type	pH	% om	CEC (meq/100 g)	PBI (DALA)	Rotational crops
	01 Oct 2012						
JSM0414-04; Ribaforada, Spain 2012	0.042 + 0.045 kg ai/ha (plot 2A); 0.042 + 0.040 kg ai/ha (plot 2B), interval 11 days, to tomato (var. H9136) 01 Oct 2012	Clay loam	8.1	2.0	153	32, 128	Wheat (var. Kilopondio (plot A and B))

Table 15 Residues of cyclaniliprole and NK-1375 in rotational wheat

Trial	Drilling of wheat (DALA)	Specimen type	Sample timing BBCH	Sample timing (DALA)	Sample timing (days after drilling)	Cyclaniliprole [mg/kg]	NK-1375 [mg/kg]
JSM0414-01 Hungary, 2012	30	Whole plant (forage)	31 - 33	219	189	< 0.01	< 0.01
	30	Whole plant (hay)	75 - 83	267	237	< 0.01	< 0.01
	30	Grain	89	297	267	< 0.01	< 0.01
	30	Straw	89	297	267	< 0.01	< 0.01
	154	Whole plant (forage)	31 - 33	219	65	< 0.01	< 0.01
	154	Whole plant (hay)	75 - 83	267	113	< 0.01	< 0.01
	154	Grain	89	297	143	< 0.01	< 0.01
	154	Straw	89	297	143	< 0.01	< 0.01
JSM0414-02 Italy, 2012	29	Whole plant (forage)	31 - 33	218	189	< 0.01	< 0.01
	29	Whole plant (hay)	75 - 83	252	223	< 0.01	< 0.01
	29	Grain	89	272	243	< 0.01	< 0.01
	29	Straw	89	272	243	0.01	< 0.01
	124	Whole plant (forage)	31 - 33	255	131	< 0.01	< 0.01
	124	Whole plant (hay)	75 - 83	271	147	< 0.01	< 0.01
	124	Grain	89	286	162	< 0.01	< 0.01
	124	Straw	89	286	162	0.01	< 0.01
JSM0414-03 Spain, 2012	32	Whole plant (forage)	31 - 33	186	154	< 0.01	< 0.01
	32	Whole plant (hay)	75 - 83	233	201	< 0.01	< 0.01
	32	Grain	89	273	241	< 0.01	< 0.01
	32	Straw	89	273	241	0.01	< 0.01
	128	Whole plant (forage)	31 - 33	242	114	0.01	< 0.01
	128	Whole plant (hay)	75 - 83	280	152	< 0.01	< 0.01
	128	Grain	89	305	177	< 0.01	< 0.01
	128	Straw	89	305	177	< 0.01	< 0.01

Study 2

This rotational crop study was conducted at six sites across the USA in 2012 to provide information on the magnitude of cyclaniliprole and its metabolite NK-1375 remaining in or on wheat at harvest following treatment of a previous growing crop with cyclaniliprole [Wiedmann and McDonald, 2013a, report IB-2012-JLW-022-01-01]. The cover crop was either tilled under or killed prior to planting the rotational crop without tillage. The field trial sites consisted of an untreated plot and one plot treated with one application of cyclaniliprole at a nominal rate of 0.3 kg ai/ha for each plant back interval (nominally 30 day, 120 day and 1 year), except for 2 plots at Oklahoma which were treated with 0.098 and 0.100 kg ai/ha. Trial characteristics are shown in Table 16.

Specimens of the target crop wheat (forage, straw and grain) were collected from the untreated and treated plots. No sampling of soil occurred for residue analysis. Wheat samples were analysed for parent and NK-1375 using HPLC-MS/MS method JSM0269. Storage intervals ranged from 28–315 days. Freezers were set to maintain ≤ -10 °C. No cyclaniliprole or NK-1375 was

detected above the LOQ in the samples from untreated plots. Control sample < 0.3 LOQ for each analyte. The concurrent recovery was within 70–120% for each analyte in each matrix

Results are shown in Table 17. The results for forage and hay were reported on as receivd basis (i.e. not calculated bbakc to dry matter content). Parent cyclaniliprole and metabolite NK-1375 were not quantifiable in any of the wheat grain samples (LOQ = 0.01 mg/kg). Wheat forage samples contained parent cyclaniliprole up to 0.022 mg/kg in the 30 and 365 day plant back and 0.028 mg/kg in the 120 day plant back interval. There were no quantifiable residues of NK-1375 in any forage samples (LOQ = 0.01 mg/kg). Parent cyclaniliprole was quantified in wheat straw up to 0.073 mg/kg in the 30 day plant back, 0.189 mg/kg in the 120 day plant back and 0.083 mg/kg in the 147 to 365 day plant back. Only two of the 36 straw samples had quantifiable residues of NK-1375, at 0.012 and 0.013 mg/kg.

Table 2 Experimental conditions for field rotational crop studies

Trial Location	Actual application; last appl date	Plot preparation	Soil type	pH	% om	CEC (meq/100 g)	PBI	Rotational crops
North Rose, NY, USA, 2012	1× 0.29 kg ai/ha to weeds; 20 April 2012 (30 d & 365 d PBI); 1x 0.30 kg ai/ha to weeds; 18 May 2012 (120 d PBI)	Plot was mowed and tilled before planting rotational crops	Silt loam	6.1	3.5	5.0	30, 120, 365	Spring wheat (var. Glenn); 30 d PBI Winter wheat (var. Pioneer 25W43); 120 d PBI Spring wheat (var. Glenn); 365 d PBI
Seven Springs, NC, USA, 2012	1× 0.30 kg ai/ha to soybean (30 d and 120 d PBI); 1x 0.31 kg ai/ha to wheat (365 d PBI); 24 Sept 2012 (30 d PBI); 2 July 2012 (120 d PBI); 20 April (365 d PBI)	Primary crop was removed before planting rotational crops	Loamy sand	5.8	0.83	3.3	30, 120, 263	Winter wheat (var. Pioneer 26R12)
Northwood, ND, USA, 2012	1× 0.31 kg ai/ha to wheat; 18 April 2012 (30 d PBI); 3 May 2012 (120 d PBI); 23 May 2012 (365 d PBI)	Primary crop was mowed, then plot was tilled before rotational crops were planted	Loam	7.2	5.1	24.8	30, 120, 366	Spring wheat (var. Faller); 30 d PBI Winter wheat (var. Jerry); 120 d PBI Spring wheat (var. Faller); 365 d PBI
Hinton, OK, USA, 2012	1× 0.10 kg ai/ha (30 d and 365 d PBI) and 1x 0.31 kg ai/ha (120 d PBI) to soybean (30 d and 120 d PBI) or to wheat (365 d PBI); 10 Oct 2012 (30 d PBI); 12 July 2012 (120 d PBI); 17 April 2012 (365 d PBI)	Plots were disked before rotational crop planting	Sandy loam	7.1	0.7	8.8	29, 119, 356	Winter wheat (var. Duster); 30 d PBI Winter wheat (var. Duster); 120 d PBI Spring wheat (var. Glenn); 365 d PBI
Porterville, CA, USA, 2012	1× 0.31 kg ai/ha to lettuce (30 d PBI) or 1x 0.30 kg ai/ha to lettuce (120 d PBI) or 1x 0.3 kg ai/ha to wheat (365 d PBI); 2 Oct 2012 (30 d PBI); 27 June 2012 (120 d PBI); 16 July 2012 (365 d PBI)	Primary crop was cultivated (30 d and 120 d PBI) before disking and planting rotational crop; plot was disked	Sandy clay loam	7.6	1.2	32.3	30, 127, 147	Winter wheat (var. Ultra)

Trial Location	Actual application; last appl date	Plot preparation	Soil type	pH	% om	CEC (meq/100 g)	PBI	Rotational crops
		and primary crop was tilled in before planting rotational crop						
Ephrata, WA, USA, 2012	1× 0.31 kg ai/ha to wheat; 17 April 2012 (30 d and 365 d PBI); 29 May 2012 (120 d PBI)	Plot was mowed and tilled before planting rotational crops	Loamy sand	8.0	0.9	12.6	30, 120, 365	Spring wheat (var. Solano); 30 d PBI Winter wheat (var. Stephens); 120 d PBI Spring wheat (var. Alpowa); 365 d PBI

Table 17 Residues of cyclaniliprole and NK-1375 in rotational wheat

Location	PBI [DALA]	DAS forage/ straw& grain	cyclaniliprole [mg/kg][a]	NK-1375 [mg/kg][a]	cyclaniliprole [mg/kg][a]	NK-1375 [mg/kg][a]	cyclaniliprole [mg/kg][a]	NK-1375 [mg/kg][a]
			Wheat forage		Wheat straw		Wheat grain	
North Rose, NY, USA, 2012	30	38/75	0.013 0.014 (0.014)	< 0.01 < 0.01 (< 0.01)	0.018 0.020 (0.019)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Seven Springs, NC, USA, 2012	30	140/232	0.011 0.012 (0.012)	< 0.01 < 0.01 (< 0.01)	0.029 0.025 (0.027)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Northwood, ND, USA, 2012	30	39/90	0.012 0.014 (0.013)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Hinton, OK, USA, 2012	29[b]	148/230	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.033 0.033 (0.033)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Porterville, CA, USA, 2012	30	125/223	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.073 0.061 (0.067)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Ephrata, WA, USA, 2012	30	56/83	0.011 0.016 (0.014)	< 0.01 < 0.01 (< 0.01)	0.035 0.057 (0.046)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
North Rose, NY, USA, 2012	120	272/306	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.026 0.022 (0.024)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Seven Springs, NC, USA, 2012	120	134/226	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.020 0.019 (0.020)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Northwood, ND, USA, 2012	120	284/342	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 0.011 (0.011)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Hinton, OK, USA, 2012	119	148/230	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.074 0.068 (0.071)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Porterville, CA, USA, 2012	127	138/224	0.024 0.028 (0.026)	< 0.01 < 0.01 (< 0.01)	0.174 0.189 (0.182)	0.012 0.013 (0.013)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Ephrata, WA, USA, 2012	120	236/310	0.022 0.016 (0.019)	< 0.01 < 0.01 (< 0.01)	0.116 0.114 (0.115)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
North Rose, NY, USA,	365	68/102	< 0.01 < 0.01	< 0.01 < 0.01	0.022 0.021	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01

Location	PBI [DALA]	DAS forage/ straw& grain	cyclaniliprole [mg/kg][a]	NK-1375 [mg/kg][a]	cyclaniliprole [mg/kg][a]	NK-1375 [mg/kg][a]	cyclaniliprole [mg/kg][a]	NK-1375 [mg/kg][a]
			Wheat forage		Wheat straw		Wheat grain	
2012			(< 0.01)	(< 0.01)	(0.022)	(< 0.01)	(< 0.01)	(< 0.01)
Seven Springs, NC, USA, 2012	263	99/156	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.021 0.019 (0.020)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Northwood, ND, USA, 2012	366	47/91	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Hinton, OK, USA, 2012	356[c]	50/84	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Porterville, CA, USA, 2012	147	147/214	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	0.044 0.027 (0.036)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)
Ephrata, WA, USA, 2012	365	44/112	0.022 0.020 (0.021)	< 0.01 < 0.01 (< 0.01)	0.080 0.083 (0.082)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)	< 0.01 < 0.01 (< 0.01)

PBI = plant back interval; DALA = days after last application; DAS = days after sowing/planting/drilling

[a] Two separate field samples were analysed; result in parenthesis is the average of the two field samples

[b] treatment with 0.098 kg ai/ha

[c] treatment with 0.100 kg ai/ha

Animal metabolism

The Meeting received information on the metabolic fate of cyclaniliprole in ruminants (lactating goats) and poultry (laying hens). The metabolism in laboratory animals was summarized and evaluated by the WHO panel of the JMPR in parallel in 2017.

Lactating goats

The metabolism of cyclaniliprole was studied in lactation goats [Kane J, 2013, report JSM0059]. One lactating goat (British Saanen) per radiolabel was dosed orally once per day, at approximately the same time each day for 5 consecutive days with a gelatin capsule containing [^{14}C-Pz]-cyclaniliprole or [^{14}C-Ph]-cyclaniliprole (radiochemical purity ≤ 7%) at a nominal rate of 10 mg ai/kg bw/day. Based on the average feed consumption/day (1.82–2.25 kg dry weight for phenyl and pyrazole label, respectively) and assuming a food consumption of 2 kg dry weight/day, the actual mean daily dose administered was 22.36 mg ai/animal/day for the phenyl or 25.15 mg ai/animal day for the pyrazole label. This was equivalent to 12.3 mg ai/kg dry feed and 11.2 mg ai/kg dry feed for the phenyl and pyrazole labelled cyclaniliprole, respectively. The age of the goats was 1–5 year for the phenyl and pyrazole label, respectively. Body weights were 69.5 and 53.0 kg at start of dosing and 63.5 and 55.0 kg at sacrifice, for the goat given the phenyl and pyrazole labelled test substance, respectively.

Milk was collected twice daily, until 23 hours after the last dose; urine and faeces were collected during 0–6 and 6–24-hour intervals after each dose, up to 23 hours after the last dose. Milk production ranged from 1.5 to 2.9 L/day. Aliquots of the PM and AM milk were mixed.

The goats were sacrificed approximately 23 hours after the administration of the final dose. Samples of liver, kidneys (both), muscle (loin and flank), fat (subcutaneous, omental, perirenal), bile, urine from bladder, rumen, reticulum (and contents), omasum, abomasum (and contents) and intestine (with contents) were taken post mortem and stored frozen at -18 °C for (2 months) prior to analysis.

Measurement of radioactivity was performed either by direct LSC (liquid and tissue samples) or by combustion/LSC (faeces, GIT contents, whole blood or whole-organ samples). The mean total recovery of the dosed radioactivity was 87.2% and 81.4% for the goats dosed with phenyl and

pyrazole radiolabelled forms, respectively. The majority of the radioactivity was recovered in faeces (68%/59% TAR, phenyl/pyrazole). The remainder of the dose was recovered in urine (5.1%/6.6% TAR phenyl/pyrazole), GI tract contents (5.4%/9.5% TAR), cage wash (0.2%/0.3% TAR), milk (0.8%/0.7% TAR), fat (3.9%/2.2% TAR), muscle (2.3%1.6% TAR), and liver (1.7%/1.4% TAR). Only very low levels were found in kidneys and bile (0.01 and < 0.01% TAR, respectively for both labels). Total recovered radioactivity in milk and tissues was 8.8%/6.0% TAR, respectively. Radioactive residues in tissues were > 0.01 mg/kg in all tissues and were extracted in order to characterised the nature of the residues.

Radioactivity levels in milk and tissues are summarised in Table 19. The highest radioactivity concentrations in edible tissues were found in the liver (1.485/1.321 mg/kg eq), fat (0.860/0.786 mg/kg eq), kidneys (0.582/0.547 mg/kg eq) followed by muscle (0.125/0.118 mg/kg eq). Total radioactive residues in milk from the phenyl label dosed goat reached a plateau concentration of approximately 0.124–0.138 mg/kg eq by 96–119 hours after dosing. Total radioactive residues in milk from the pyrazole label dosed goat reached a plateau concentration of approximately 0.081–0.091 mg/kg eq following 72 hours after dosing.

Residue levels in cream and skimmed milk derived from combined PM and AM milk samples (24 hour samples) are summarised in Table 18. Concentrations of the residues were highest in milk collected in the afternoon, indicating a rapid elimination.

Table 18 Total radioactive residues in afternoon and morning collections and in daily pooled milk

Milk Sample Time Point	TRR (mg/kg eq)					
	[^{14}C-Ph]-cyclaniliprole			[^{14}C-Pz]-cyclaniliprole		
	afternoon	morning	daily pooled	afternoon	morning	daily pooled
0–24 hours	0.055	0.038	0.045	0.055	0.051	0.053
24–48 hours	0.111	0.057	0.074	0.065	0.103	0.081
48–72hours	0.119	0.078	0.090	0.118	0.072	0.086
72–96 hours	0.190	0.087	0.138	0.119	0.073	0.087
96–119 hours	0.171	0.093	0.124	0.115	0.076	0.091

Homogenized liver and kidney samples were extracted with acetonitrile (two or three times) and acetonitrile:water (1:1 v/v) followed by centrifugation. The first extract was taken for LSC directly, the second was dried and re-suspended in acetonitrile:water (1:1, v/v) prior to LSC and HPLC/TLC. Post-extraction solids were dried and then incubated with protease enzymes in 0.01 M phosphate buffer (at *ca*, pH 7.5, 18 hours at 37 °C). Following incubation, samples were then extracted with acetonitrile and centrifuged, with the supernatant taken for LSC. The enzyme-treated solid residues were incubated with 1M HCl (18 hours at 37 °C), then extracted twice with water and centrifuged, with the supernatant taken for LSC. The acid-treated solid residues were incubated (18 hours at 37 °C) with 1M NaOH then extracted with acetone and centrifuged, with the supernatant taken for LSC and HPLC/TLC. The final solid residues were dried and portions taken for combustion/LSC.

Homogenized fat samples were extracted with hexane and centrifuged. Aliquots of supernatant were taken for LSC. Remaining supernatant was partitioned with acetonitrile and aliquots of each layer taken for LSC. The PES was then further extracted twice with acetonitrile, and aliquots either taken for LSC or analysed HPLC/TLC. The final solid residues were dried and portions taken for solubilisation/LSC.

Homogenised muscle samples (pools of flank and loin muscle) were extracted with acetonitrile (two or three times) and acetonitrile:water (1:1 v/v) followed by centrifugation. Aliquots of supernatant were taken for LSC or dried and re-suspended for analysis by HPLC/TLC. The PES were dried and portions taken for solubilisation/LSC.

Pooled whole-milk samples (phenyl label: days 4–5, pyrazole label: days 2–5) were initially subdivided into a whole-milk subsample and a sub-sample for centrifugation to separate the milk fat

from the aqueous fraction. For the whole milk and milk-fat fractions, the sub-samples were initially aliquoted for direct LSC and then extracted and centrifuged three times with hexane and then acetonitrile. Aliquots of the supernatants were taken for LSC. The hexane extract was then further partitioned with acetonitrile. The acetonitrile layer was pooled with acetonitrile extracts and aliquots of supernatant taken for LSC or dried and re-suspended for analysis by HPLC/TLC.

Aqueous-fraction milk samples were initially aliquoted for direct LSC and then extracted and centrifuged with acetonitrile. Aliquots of supernatant were taken for LSC or dried and re-suspended for analysis by HPLC/TLC. The solid residue was dried and portions taken for solubilisation/LSC.

Extractability of the radioactive residues with neutral solvents ranged from 84% TRR in kidney to 99.6% in fat. Sequential treatment of PES with protease, acid and base released an additional 2.8/3.1, 0.8/8.3, 8.0/2.8% TRR, respectively with the ^{14}C-Ph and ^{14}C-Pz label in liver and 5.4, 4.1, and 5.7% TRR, respectively in kidney with the ^{14}C-Ph label (see Table 19 and Table 20). Identified radio-components present in extracted residues (combined solvent extracts and radioactivity released from PES by harsh treatment) are summarised in Tables 20–Table 23.

Residues were identified by co-chromatography with authentic reference standards for parent, YT-1284, NSY-28, and NSY-27.

Initial radio-component profiles of fractions of liver and whole milk samples were produced within 125 days of sacrifice. Re-analysis of muscle, liver, kidney, fat and whole milk fractions after storage of the extracts for periods up to 3 months under frozen conditions showed that no major changes occurred in the proportion of the components. The results indicate stability of the residues from initial analysis until final analysis.

Table 19 Total Radioactive Residues and extractability in samples from goats treated with [^{14}C]-cyclaniliprole

Sample	[^{14}C-Ph]-cyclaniliprole Extracted Radioactivity		[^{14}C-Pz]-cyclaniliprole Extracted Radioactivity	
	%TRR	mg/kg eq	%TRR	mg/kg eq
Whole milk (pool day 4–5 phenyl and 2–5 pyrazole)	98.2	0.129	98.2	0.081
Whole milk PES	1.8	0.002	1.8	0.001
Whole milk total	100	0.131	100	0.082
Liver, normal solvents	85.8	1.274	85.5	1.129
- protease treatment	*2.8*	*0.042*	*3.1*	*0.041*
- acid treatment	*0.8*	*0.012*	*8.3*	*0.110*
- base treatment	*8.0*	*0.119*	*2.8*	*0.037*
Liver total extracted[b]	97.4[b]	1.447[b]	99.6[b]	1.316[b]
Liver PES[c]	2.6	0.038	0.4	0.005
Liver total	100	1.485	100	1.321
Kidney, normal solvents	84	0.489	92.9	0.508
- protease treatment	*5.4*	*0.031*	-	-
- acid treatment	*4.1*	*0.024*	-	-
- base treatment	*5.7*	*0.033*	-	-
Kidney total extracted[b]	99.2[b]	0.578[b]	n.a.	n.a.
Kidney PES[c]	0.8	0.004	7.1	0.039
Kidney total	100	0.582	100	0.547
Composite muscle[d]	95.6	0.114	96.8	0.116
Composite muscle[d] PES	4.4	0.005	3.2	0.004
Composite muscle[d] total	100%	0.119	100	0.120
Composite fat[e]	99.6	0.877	99.0	0.696
Composite fat[e] PES	0.4	0.003	1.0	0.007
Composite fat[e] total	100	0.880	100	0.703

[a] TRR in mg/kg eq calculated from radioactivity extracted and radioactivity in the PES.

[b] Including extracted radioactivity after protease, acid and base treatment, individual values presented in italics.

[c] Radioactivity remaining in the solids after solvent extraction and subsequent treatment with acid, base and enzymes

[d] Pool of flank and loin muscle to produce a single composite sample.

[e] Pool of subcutaneous, omental and perirenal fat produce a single composite sample.

Table 20 Characterisation and identification of components in samples from goats treated with [^{14}C]-cyclaniliprole

	[^{14}C-Ph]-cyclaniliprole					[^{14}C-Pz]-cyclaniliprole				
	Milk %TRR	Liver %TRR[c]	Kidney %TRR[c]	Muscle %TRR	Fat %TRR	Milk %TRR	Liver %TRR[c]	Kidney %TRR	Muscle %TRR	Fat %TRR
Total analysed	98.2 (0.129)	97.4 (1.447)	99.2 (0.578)	95.6 (0.114)	99.6 (0.877)	98.2 (0.081)	99.6 (1.316)	92.9 (0.508)	96.8 (0.116)	99.0 (0.696)
Parent	71.4 (0.94)	32.7 (0.485)	29.7 (0.173)	43.8 (0.052)	76.4 (0.673)	58.4 (0.048)	30.1 (0.397)	19.0 (0.10)	22.7 (0.027)	44.3 (0.311)
YT-1284	11.2 (0.015)	13.5 (0.202)	13.1 (0.076)	16.9 (0.020)	6.0 (0.053)	21.2 (0.017)	18.6 (0.246)	10.7 (0.058)	17.3 (0.021)	5.7 (0.040)
NSY-28	5.2 (0.007)	25.4 (0.376)	36.1 (0.210)	27.0 (0.032)	8.0 (0.070)	9.4 (0.008)	32.0 (0.422)	53.2 (0.291)	45.9 (0.055)	43.4 (0.305)
NSY-27	1.4 (0.002)	4.7 (0.070)	4.3 (0.025)	nd -	1.5 (0.013)	2.4 (0.002)	1.5 (0.020)	2.6 (0.014)	nd -	nd -
Total identified (parent and metabolites)	89.2 (0.118)	76.3 (1.134)	83 (0.484)	87.7 (0.104)	91.9 (0.809)	91.4 (0.075)	82.2 (1.085)	85.5 (0.467)	85.9 (0.103)	93.4 (0.656)
Met-1 (Rt~4/5 min)	0.4 (0.001)	1.7 (0.025)	2.5 (0.014)	2.4 (0.003)	0.2 (0.002)	0.3 (< 0.000)	2.5 (0.035)	1.4 (0.008)	2.4 (0.003)	0.9 (0.006)
Met-2 (Rt~6/7 min)	nd -	1.2 (0.018)	0.8 (0.005)	nd -	nd -	nd -	1.9 (0.025)	nd -	nd -	nd -
Met-3 (Rt~9 min)	nd -	0.9 (0.014)	0.9 (0.005)	nd -	nd -	2.7 (0.002)	3.3 (0.44)	1.4 (0.008)	3.5 (0.004)	nd -
Met-4 (Rt~14.5 min)	nd -	0.4 (0.005)	0.6 (0.003)	nd -	nd -	nd -	1.9 (0.025)	nd -	nd -	nd -
Met-6 (Rt~20 min.)	3.7 (0.005)	6.4 (0.094)	5.0 (0.029)	3.2 (0.004)	4.4 (0.039)	nd -	2.0 (0.024)	nd -	nd -	nd -
Met-10 (Rt~33/34 min)	nd -	nd -	0.1 (0.001)	nd -	nd -	nd -	nd -	nd -	1.0 (0.001)	nd -
Met-11 (Rt~37 min.)	nd -	nd -	0.1 (< 0.001)	nd -	nd -	nd -	nd -	nd -	nd -	nd -
Others	4.9 (0.006)	10.6 (0.157)	6.3 (0.036)	2.45 (0.003)	3.2 (0.028)	3.8 (0.003)	4.7 (0.064)	4.6 (0.025)	4.0 (0.005)	4.8 (0.033)
Total identified or characterised	98.2 (0.129)	97.4 (1.447)	99.2 (0.578)	95.6 (0.114)	99.6 (0.877)	98.2 (0.081)	99.6 (1.316)	92.9 (0.508)	96.8 (0.116)	99.0 (0.696)
PES[b]	1.8 (0.002)	2.6 (0.038)	0.8 (0.004)	4.4 (0.005)	0.4 (0.003)	1.8 (0.001)	0.4 (0.005)	7.1 (0.039)	3.2 (0.004)	1.0 (0.007)
Total[c]	100 (0.131)	1.485	0.582	100 (0.119)	100 (0.880)	100 (0.082)	1.321	0.547	100 (0.120)	100 (0.703)

nd = not detected (< 0.001 mg/kg eq); ( ) values between brackets are levels in mg/kg eq; (Rt~) = retention time in miutes; PES: post extraction solids

[a] Results are expressed as the total of the solvent extraction and protease and acid and base treatments. Metabolites found in the different treatment fractions are summarised in Table and Table .

[b] Radioactivity remaining in the solids after solvent extraction and subsequent teratment with acid, base and enzymes

[c] TRR determined by summation of radioactivity present in the extracts and solids following solvent extraction

Table 21 Proportion of radio-active components in different treated fractions of liver after administration of cyclaniliprole

	[^{14}C-Ph]-cyclaniliprole					[^{14}C-Pz]-cyclaniliprole				
	solvent	protease	acid	base	Total	solvent	protease	acid	base	Total
Total analysed	85.8 (1.274)	2.8 (0.042)	0.8 (0.012)	8.0 (0.119)	97.4 (1.447)	85.5 (1.129)	3.1 (0.041)	8.3 (0.110)	2.8 (0.037)	99.6 (1.316)
Parent	31.6 (0.469)	0.5 (0.008)	nd -	0.6 (0.009)	32.7 (0.486)	30.1 (0.397)	nd -	nd -	nd -	30.1 (0.397)
YT-1284	12.1 (0.180)	0.3 (0.005)	< 0.1 (0.001)	1.1 (0.016)	13.5 (0.202)	16.9 (0.224)	0.4 (0.005)	1.3 (0.017)	nd -	18.6 (0.246)
NSY-28	24.2 (0.359)	0.4 (0.005)	< 0.1 (0.001)	0.8 (0.012)	25.4 (0.376)	31.45 (0.414)	0.3 (0.004)	nd -	0.3 (0.004)	32.0 (0.422)
NSY-27	4.3 (0.064)	nd -	< 0.01 (0.001)	0.4 (0.006)	4.7 (0.070)	1.5 (0.020)	nd -	nd -	nd -	1.5 (0.020)
Met-1 (Rt~4/5 min)	0.3 (0.004)	0.2 (0.003)	0.2 (0.003)	1.0 (0.015)	1.7 (0.025)	0.4 (0.006)	0.4 (0.006)	1.2 (0.016)	0.05 (0.007)	2.5 (0.035)
Met-2 (Rt~6/7 min)	nd -	0.1 (0.002)	0.2 (0.003)	0.9 (0.013)	1.2 (0.018)	nd -	0.3 (0.004)	1.6 (0.021)	nd -	1.9 (0.025)
Met-3 (Rt~9 min)	nd -	0.2 (0.030)	0.1 (0.002)	0.6 (0.009)	0.9 (0.014)	1.1 (0.015)	0.5 (0.006)	1.6 (0.022)	0.1 (0.001)	3.3 (0.44)
Met-4 (Rt~14.5 min)	nd -	0.3 (0.004)	0.1 (0.001)	nd -	0.4 (0.005)	nd -	0.3 (0.004)	1.1 (0.014)	0.5 (0.007)	1.9 (0.025)
Met-6 (Rt~20 min.)	4.8 (0.071)	0.4 (0.005)	< 0.1 (0.001)	1.2 (0.017)	6.4 (0.094)	nd -	0.6 (0.007)	0.8 (0.010)	0.6 (0.007)	2.0 (0.024)
Met-10 (Rt~33/34 min)	nd -	nd -	nd -	nd -	nd -	nd -	nd -	nd -	nd -	nd -
Met-11 (Rt~37 min.)	nd -	nd -	nd -	nd -	nd -	nd -	nd -	nd -	nd -	nd -
Others	8.6 (0.127)	0.4 (0.006)	0.1 (0.002)	1.5 (0.022)	10.6 (0.157)	4.0 (0.053)	0.3 (0.004)	0.7 (0.010)	0.5 (0.007)	4.7 (0.064)
Total	85.8 (1.274)	2.8 (0.042)	0.8 (0.012)	8.0 (0.119)	97.4 (1.447)	85.5 (1.129)	3.1 (0.041)	8.3 (0.110)	2.8 (0.037)	99.6 (1.316)

Table 22 Proportion of radio-active components in different treated fractions of kidney after administration of cyclaniliprole

	[^{14}C-Ph]-cyclaniliprole				
	solvent	protease	acid	base	Total
Total analysed	84.0 (0.489)	5.4 (0.031)	4.1 (0.024)	5.7 (0.033)	99.2 (0.578)
Parent	28.3 (0.165)	0.4 (0.002)	0.2 (0.001)	0.8 (0.005)	29.7 (0.173)
YT-1284	10.5 (0.061)	0.6 (0.003)	0.9 (0.005)	1.1 (0.007)	13.1 (0.076)
NSY-28	34.7 (0.202)	0.5 (0.003)	0.2 (0.001)	0.7 (0.004)	36.1 (0.210)
NSY-27	2.4 (0.014)	0.5 (0.003)	1.0 (0.006)	0.4 (0.002)	4.3 (0.025)
Met-1 (Rt~4/5 min)	1.2 (0.007)	0.4 (0.002)	0.4 (0.002)	0.5 (0.003)	2.5 (0.014)
Met-2 (Rt~6/7 min)	nd -	0.4 (0.002)	0.3 (0.002)	0.1 (0.001)	0.8 (0.005)
Met-3 (Rt~9 min)	nd -	0.2 (0.001)	0.4 (0.002)	0.3 (0.002)	0.9 (0.005)
Met-4 (Rt~14.5 min)	nd -	0.4 (0.002)	0.2 (0.001)	nd -	0.6 (0.003)
Met-6 (Rt~20 min.)	3.2 (0.019)	0.7 (0.004)	0.4 (0.002)	0.7 (0.004)	5.0 (0.029)
Met-10 (Rt~33/34 min)	nd -	0.1 (0.001)	nd -	nd -	0.1 (0.001)

	[^{14}C-Ph]-cyclaniliprole				
	solvent	protease	acid	base	Total
Total analysed	84.0 (0.489)	5.4 (0.031)	4.1 (0.024)	5.7 (0.033)	99.2 (0.578)
Met-11 (Rt~37 min.)	nd -	0.1 (0.001)	nd -	nd -	0.1 (< 0.001)
Others	3.8 (0.022)	1.2 (0.007)	0.3 (0.001)	1.0 (0.006)	6.3 (0.036)
Total	84.0 (0.489)	5.4 (0.031)	4.1 (0.024)	5.7 (0.033)	99.2 (0.578)

Table 23 Metabolites found in pooled milk in fat and aqueous fractions

Milk Sample	%TRR (mg/kg eq)					
	[^{14}C-Ph]-cyclaniliprole (pooled milk from days 4-5)			[^{14}C-Pz]-cyclaniliprole (pooled milk from days 2-5)		
	whole milk	milk fat	milk aqueous	whole milk	milk fat	milk aqueous
parent	71.4 (0.094)	79.5 (0.751)	41.9 (0.014)	58.4 (0.048)	72.1 (0.527)	30.9 (0.008)
YT-1284	11.2 (0.015)	2.1 (0.019)	24.8 (0.009)	21.2 (0.017)	6.4 (0.047)	30.4 (0.008)
NSY-28	5.2 (0.007)	6.7 (0.063)	7.9 (0.003)	9.4 (0.008)	8.6 (0.063)	11.8 (0.003)
NSY-27	1.4 (0.002)	0.8 (0.007)	5.3 (0.002)	2.4 (0.002)	-	3.7 (0.001)

Laying hens

The metabolism of cyclaniliprole was studies in laying hens [Jones, 2013, report JSM0060]. Ten laying hens (*Lohman Lite*, 29–39 weeks old, weighing 1.7–2.5 kg) were dosed orally with either ^{14}C-phenyl labelled or ^{14}C-pyrazole-labelled cyclaniliprole. The hens were dosed once daily during 14 days via capsules containing 1.5 mg and 1.4 mg phenyl- or pyrazole-labelled cyclaniliprole, respectively, corresponding to a nominal concentration of 10 mg/kg in the diet. With a total dry feed intake of 1678–2153 g, this lead to an actual rate of 11.3 and 10.8 mg/kg phenyl- or pyrazole-labelled cyclaniliprole in dry feed, respectively.

Excreta and eggs were collected twice daily during the test period until 12 hours after the last dose on day 14. Twelve hours after the last treatment, the hens were sacrificed and tissues (fat, muscle, skin, liver) were collected. Eggs were separated into egg yolk and egg white.

Samples were stored at -18 °C. The last analysis occurred 196 days after sacrifice. Re-analysis of the ^{14}C-pyrazole labelled samples of liver, muscle, fat, and egg 51 days after the last analysis showed that no major changes had occurred in the proportions of the components during storage of extract for periods of approximately six months under frozen conditions.

All samples were analysed for total radioactivity by liquid scintillation counting (LSC) (cage wash and plasma), solubilisation/LSC (pooled egg, partially formed egg and pooled tissues) or combustion/LSC (excreta and liver). Residues were expressed as cyclaniliprole equivalents. Overall recoveries were 95.5 and 97.5% of the cumulative dose of ^{14}C-phenyl- or ^{14}C-pyrazole-labelled cyclaniliprole. Radioactivity in excreta and cage washes accounted for 91.7% and 92.9% TAR and 1.0 and1.4% TAR, respectively for both labels. Radioactivity remaining in tissues and at sacrifice accounted for 0.8 and 0.7% of the administered dose. Radioactivity in eggs accounted for 2.0 and 2.5% of the dose for the respective labels.

Total radioactive residues in eggs are shown in Table 24. The residues in eggs ranged between 0.200–0.761 and 0.175–0.964 mg/kg cyclaniliprole equivalents for ^{14}C-phenyl- or ^{14}C-pyrazole-labelled samples and reached a plateau after approximately 9 days after start of dosing.

Concentrations of the residues were highest in eggs collected in the afternoon, indicating a rapid elimination.

Table 24 Total radioactive residues in eggs

Time (after 1st dose)	[^{14}C-Ph]-cyclaniliprole (mg/kg eq)			[^{14}C-Pz]-cyclaniliprole (mg/kg eq)		
Days	afternoon	morning	daily pooled	afternoon	morning	daily pooled
pre-dose	na	< 0.001	-	na	< 0.001	na
1/2	ns	0.200	0.2	< 0.001	0.237	0.175
2/3	0.925	0.266	0.441	0.093	0.265	0.247
3/4	0.957	0.355	0.473	1.062	0.327	0.486
4/5	1.170	0.526	0.650	0.757	0.446	0.551
5/6	ns	0.469	0.469	0.669	0.625	0.642
6/7	1.200	0.509	0.573	1.084	0.531	0.964
7/8	1.415	0.536	0.621	0.986	0.537	0.669
8/9	1.436	0.666	0.748	1.103	0.728	0.829
9/10	1.451	0.573	0.686	0.861	0.564	0.623
10/11	1.017	0.640	0.719	0.894	0.613	0.668
11/12	1.626	0.539	0.755	0.912	0.670	0.729
12/13	1.231	0.526	0.602	0.570	0.761	0.744
13/14	1.227	0.703	0.761	ns	0.613	0.613
14	1.258	na	1.258	0.377	na	0.377

na = not applicable; ns= no sample

Total radioactive residues found in tissues are shown in Table 25. Residues were highest in liver (1.659 and 1.466 mg/kg eq). Total radioactivity residues in fat (abdominal and subcutaneous), muscle (breast and leg) and skin accounted for 0.056–0.347 and 0.058–0.304 mg/kg eq, respectively.

Eggs were sequentially extracted with hexane and acetonitrile (2 × ^{14}C-phenyl label, 3 × ^{14}C-pyrazole label) and acetonitrile:water (1:1 v/v × 1). A subsample of the PES (^{14}C-phenyl label only) was incubated with protease enzymes in 0.067 M phosphate buffer at pH 7.0 (16 hrs, 37 °C), followed by extraction with acetonitrile and acetonitrile:water (1:1 v/v, × 1). A subsample (^{14}C-label only) of PES was incubated with protease enzymes in phosphate buffer at pH 7.0 (16 hrs, 37 °C). Following incubation, the residue was extracted with acetonitrile (× 1) and acetonitrile:water (1:1 v/v, × 1). The protease treated egg PES was incubated with 1 M HCl (16 hrs, 37 °C) followed by extraction with acetonitrile and then acetonitrile:water (1:1 v/v, ×1) and taken for radioassay. The solids remaining after acid treatments were incubated with 1M NaOH (16 hrs, 37 °C), followed by extraction with acetonitrile and acetonitrile:water (1:1 v/v, × 1).

Liver homogenate was extracted with acetonitrile (× 3) followed by extraction with acetonitrile:water (1:1 v/v, × 2). The same analytical procedures were applied to the liver PES as for the eggs up until the incubation with NaOH and extraction with acetonitrile and acetonitrile:water. After taking aliquots for radioassay, the extracts were combined and pH adjusted to 2. The sample was partitioned with ethyl acetate (× 3) and extracts were combined.

The hexane extract from the skin homogenate and equal portions of the subcutaneous and abdominal fat homogenates were partitioned with acetonitrile (× 3). The PES remaining were further extracted with acetontrile (2 × ^{14}C-phenyl, 3× ^{14}C-pyrazole) and acetonitrile:water (1:1 v/v × 1). A proportionate pool was prepared by combining portions of these extracts with the acetonitrile extracts.

Equal portions of the leg and breast muscle homogenates were pooled and extracted with acetonitrile (× 3) followed by acetonitrile:water (1:1 v/v, × 2). The same procedures as for egg PES were followed to obtain protease, acid and base treated extracts.

Liver, eggs, skin, fat and muscle were extracted with neutral solvents and analysed chromatographically within two months of sacrifice.

Extractability of the radioactive residues with neutral solvents > 76% TRR. Sequential treatment of PES with protease, acid and base in liver, muscle and eggs released an additional 9.7–

22.8% TRR, (see Tables 25–Table 27). Identified radio-components present in extracted residues (combined solvent extracts and radioactivity released from PES by harsh treatment) are summarised in Tables 26–Table 27).

Table 25 Total Radioactive residues and extractability in samples from laying hens treated with [^{14}C]-cyclaniliprole

Sample	Extracted Radioactivity [^{14}C-Ph]-cyclaniliprole		Extracted radioactivity [^{14}C-Pz]-cyclaniliprole	
	%TRR	mg/kg eq	%TRR	mg/kg eq
Liver, total neutral[b]	81.5	1.352	76.3	1.119
- protease treatment	*9.3*	*0.154*	*6.8*	*0.100*
- acid treatment	*2.8*	*0.046*	*13.1*	*0.192*
- base treatment	*6.4*	*0.106*	*3.8*	*0.056*
Liver, total extracted[c]	100[c]	1.659[c]	100[c]	1.466[c]
Liver, solids remaining[d]	< 0.1	< 0.001	< 0.1	< 0.001
Liver, total[a]	100	1.629	100	1.466
Egg, total neutral[be]	90.4	0.628	93.6	0.625
- protease treatment	*6.9*	*0.048*	-	-
- acid treatment	*2.8*	*0.019*	-	-
- base treatment	*< 0.1*	*< 0.001*	-	-
Egg, total extracted[ce]	100[c]	0.695[c]	n.a.	n.a.
Egg, solids remaining[d]	< 0.1	< 0.001	6.3	0.042
Egg, total[a]	100.1	0.695	99.9	0.668
Muscle[f] total neutral	90.7	0.065	84.9	0.057
- protease treatment	-	-	*6.1*	*0.004*
- acid treatment	-	-	*4.5*	*0.003*
- base treatment	-	-	*4.6*	*0.003*
Muscle, total extracted[c]	n.a.	n.a.	100	0.067
Muscle[f] PES	9.3	0.007	< 0.1	< 0.001
Muscle[f] total	100	0.072	100	0.067
Fat[g] extracted	98.2	0.336	98.3	0.265
Fat[g] PES	1.8	0.006	1.7	0.005
Fat[g] total	100	0.342	100	0.269
Skin, extracted	90.7	0.244	92.2	0.280
Skin, PES	9.3	0.025	7.8	0.024
Skin total	100	0.269	100	0.304

[a] TRR calculated by summation of radioactivity after solvent extraction and subsequent teratment with acid, base and enzymes and remaining solids.

[b] Sum of neutral extracts (hexane and acetonitrile fractions)

[c] Sum of neutral solvent extractions and protease, acid and base treatment fractions, individual values presented in italics.

[d] Post extraction solids and solids remaining are the solid left after protease, acid and base treatment.

[e] Pool of day 9-15 eggs (phenyl label) or day 9-14 eggs (pyrazole label)

[f] Pool of (equal portions of) breast and leg muscle (0.088 and 0.075 mg/kg eq for leg muscle with the respective labels and 0.056 and 0.058 mg/kg eq for breast muscle for the respective labels)

[g] Pool of (equal portions of) abdominal and subcutaneous fat (0.347 and 0.276 mg/kg eq for abdominal fat with the respective labels and 0.337 and 0.262 mg/kg eq for subcutaneous fat for the respective labels)

Table 26 Amount and nature of residues in hen tissues treated with cyclaniliprole as % TRR (and in mg/kg equivalents)

Label	[^{14}C-Ph]-cyclaniliprole					[^{14}C-Pz]-cyclaniliprole				
Tissues	Egg[ab]	Fat[c]	Skin	Muscle[d]	Liver	Egg[a]	Fat[c]	Skin	Muscle[bd]	Liver[b]
% TRR[e]	100	100 (0.342)	100	100	100	100	100	100	100	100
(mg/kg eq)	-0.695		-0.269	-0.072	-1.659	-0.668	-0.269	-0.304	-0.067	-1.466
- parent	21	43.7	25.8	15.5	4.4	23.4	58.5 (0.158)	29.7	9.7 (0.006)	11.5
	-0.146	-0.149	-0.069	-0.011	-0.073	-0.156		-0.09		-0.17

Label	[^{14}C-Ph]-cyclaniliprole					[^{14}C-Pz]-cyclaniliprole				
Tissues	Egg[ab]	Fat[c]	Skin	Muscle[d]	Liver	Egg[a]	Fat[c]	Skin	Muscle[bd]	Liver[b]
- YT-1284	6.6	10.6	11.1	27.7	8.6	4.0 (0.027)	9.2 (0.025)	10	16.4	10.8 (0.160)
	-0.046	-0.036	-0.03	-0.02	-0.142			-0.03	-0.011	
- NSY-28	54.4	27.3	38.3	26.6	63.2	62.8	25.5	47.2	48.5	55.7 (0.816)
	-0.378	-0.093	-0.103	-0.019	(1.049	-0.419	-0.069	-0.143	-0.033	
- NSY-27	2.3	4.2	5.2	6.2	< 0.5	< 0.3 (< 0.002)	<1.4 (< 0.004)	0.5 (0.002)	1.5	< 0.6 (< 0.008)
	-0.016	-0.014	-0.014	-0.004	(< 0.007)				-0.001	
Total identified	84.3 (0.586)	85.8	80.4	76	76.2	90.2 (0.602)	93.2	87.4	76.1	78
		-0.292	-0.216	-0.054	-1.264		-0.252	-0.265	-0.051	-1.146
Hn-A	5.2	< 0.7	< 0.2	3.2	5.4	0.5	<1.4	< 0.4	< 0.9	6.4
(Rt~4-8 min)	-0.036	(< 0.002)	(< 0.001)	-0.002	-0.088	-0.003	(< 0.004)	(< 0.001)	(< 0.001)	-0.094
Hn-B	0.1	< 0.7	< 0.2	< 0.9	2.8	< 0.3	<1.4	< 0.4	< 0.9	3.2
(Rt~9-11 min)	-0.001	(< 0.002)	(< 0.001)	-0.001	-0.048	(< 0.002)	(< 0.004)	(< 0.001)	(< 0.001)	-0.048
Hn-C	< 0.5	< 0.7	< 0.2	< 0.9	2.8	< 0.3	<1.4	< 0.4	< 0.9	1.3
(Rt~11-13 min)	(< 0.003)	(< 0.002)	(< 0.001)	-0.001	-0.047	(< 0.002)	(< 0.004)	(< 0.001)	(< 0.001)	-0.018
Hn-D	0.5	< 0.7	< 0.2	< 0.9	1.6	< 0.3	<1.4	1.4	3.2	1.9
(Rt~14-16 min)	-0.003	(< 0.002)	(< 0.001)	-0.001	-0.028	(< 0.002)	(< 0.004)	-0.004	-0.002	-0.027
Hn-E	5.2	1.5	1.7	7.6	4.5	2.2	3.4	2.4	5.6	4.2
(Rt~17-19 min)	-0.036	-0.005	-0.005	-0.005	-0.074	-0.015	-0.009	-0.007	-0.004	-0.061
Hn-I (Rt~29-30 min)	2.9	7.1	7.4	3.9	1.6	0.4	<1.4	< 0.4	< 0.9	1.7
	-0.02	-0.024	-0.02	-0.003	-0.025	-0.003	(< 0.004)	(< 0.001)	(< 0.001)	-0.024
Others	0.2	0.7	< 0.2	< 0.9	2.5	< 0.3	<1.4	< 0.4	< 0.9	0.1
	-0.001	-0.002	(< 0.001)	(< 0.001)	-0.041	(< 0.002)	(< 0.004)	(< 0.001)	(< 0.001)	-0.002
Total analysed[e]	98.4	95.1	89.6	90.7	97.4	93.3	96.6	91.2	84.9	99.1
	-0.683	-0.325	-0.241	-0.065	-1.615	-0.623	-0.26	-0.277	-0.057	-1.454
Unanalysed fraction[f]	1.7	3.1	1.1	nd	2.6	0.3	1.7	1	15.1	0.9
	-0.012	-0.011	-0.003	-	-0.043	-0.002	-0.005	-0.003	-0.01	-0.013
Total	100 (0.695)	98.2	90.7	90.7	100	93.6	98.3	92.2 (0.280)	100	100
		-0.336	-0.244	-0.065	-1.659	-0.625	-0.265		-0.067	-1.466
PES	< 0.01	1.8	9.3	9.3	< 0.1	6.3 (0.042)	1.7	7.8	< 0.1	< 0.1
	(< 0.001)	-0.006	-0.025	-0.07	(< 0.001)		-0.005	-0.024	(< 0.001)	(< 0.001)
% TRR[g]	100	100 (0.342)	100	100	100	100	100	100	100	100
(mg/kg eq)	-0.695		-0.269	-0.072	-1.659	-0.668	-0.269	-0.304	-0.067	-1.466

PES = Post-extracted solids

[a] Pool of day 9–15 eggs (phenyl label) or day 9–14 eggs (pyrazole label)

[b] Results are expressed as the total of the solvent extraction and protease and acid and base treatments. Metabolites found in the different treatment fractions are summarised in Table 27 and Table 28.

[c] Pool of (equal portions of) abdominal and subcutaneous fat (0.347 and 0.276 mg/kg eq for abdominal fat with the respective labels and 0.337 and 0.262 mg/kg eq for subcutaneous fat for the respective labels)

[d] Pool of (equal portions of) breast and leg muscle (0.088 and 0.075 mg/kg eq for leg muscle with the respective labels and 0.056 and 0.058 mg/kg eq for breast muscle for the respective labels)

[e] Total of identified and characterised compounds

[f] Un-analysed fraction for egg is hexane or unidentified; aqueous fraction for liver; un-analysed fraction is hexane fraction for fat and skin; for muscle the unanalysed fraction consists of the protease, acid and base fractions for the ^{14}C-Pz label.

[g] TRR calculated by summation of extracted radioactivity and post-extracted solids (PES)

Table 27 Proportion of radio-active components in different treated fractions of liver after administration of cyclaniliprole

Label	[^{14}C-Ph]-cyclaniliprole					[^{14}C-Pz]-cyclaniliprole				
	solvent	protease	acid	base	Total	solvent	protease	acid	base	Total
Total analysed	81.5 (1.352)	9.3 (0.154)	2.8 (0.046)	3.8 (0.063)	97.4 (1.615)	76.3 (1.119)	6.8 (0.100)	13.1 (0.192)	2.9 (0.043)	99.1 (1.454)
- parent	3.4 (0.057)	< 0.1 (< 0.001)	< 0.1 (< 0.001)	1.0 (0.016)	4.4 (0.073)	4.8 (0.070)	< 0.1 (< 0.001)	5.5 (0.081)	1.2 (0.019)	11.5 (0.170)
- YT-1284	5.9 (0.097)	0.5 (0.008)	1.1 (0.018)	1.1 (0.019)	8.6 (0.142)	8.2 (0.121)	0.3 (0.005)	2.3 (0.034)	< 0.2 (< 0.002)	10.8 (0.160)
- NSY-28	62.6 (1.038)	< 0.1 (< 0.001)	< 0.1 (< 0.001)	0.6 (0.011)	63.2 (1.049)	54.9 (0.805)	< 0.1 (< 0.001)	0.5 (0.007)	0.3 (0.004)	55.7 (0.816)
- NSY-27	< 0.2 (< 0.004)	< 0.1 (< 0.001)	< 0.1 (< 0.001)	< 0.1 (< 0.001)	< 0.5 (< 0.007)	< 0.2 (< 0.003)	< 0.1 (< 0.001)	< 0.1 (< 0.002)	< 0.2 (< 0.002)	< 0.6 (< 0.008)
Hn-A (Rt~4-8 min)	< 0.2 (< 0.004)	3.5 (0.058)	1.4 (0.022)	0.5 (0.008)	5.4 (0.088)	1.2 (0.018)	2.5 (0.037)	2.1 (0.031)	0.6 (0.008)	6.4 (0.094)
Hn-B (Rt~9-11 min)	0.4 (0.007)	2.2 (0.037)	0.2 (0.004)	< 0.1 (< 0.001)	2.8 (0.048)	0.9 (0.013)	1.6 (0.024)	0.7 (0.011)	< 0.2 (< 0.002)	3.2 (0.048)
Hn-C (Rt~11-13 min)	1.3 (0.022)	1.3 (0.022)	0.1 (0.002)	0.1 (0.001)	2.8 (0.047)	0.3 (0.004)	1.0 (0.014)	< 0.1 (< 0.02)	< 0.2 (< 0.002)	1.3 (0.018)
Hn-D (Rt~14-16 min)	0.8 (0.014)	0.7 (0.012)	< 0.1 (< 0.001)	0.1 (0.002)	1.6 (0.028)	0.8 (0.012)	0.5 (0.008)	0.4 (0.005)	0.2 (0.002)	1.9 (0.027)
Hn-E (Rt~17-19 min)	3.3 (0.054)	0.9 (0.015)	< 0.1 (< 0.001)	0.3 (0.005)	4.5 (0.074)	2.1 (0.030)	0.7 (0.010)	0.8 (0.011)	0.6 (0.010)	4.2 (0.061)
Hn-I (Rt~29-30 min)	1.5 (0.024)	< 0.1 (< 0.001)	< 0.1 (< 0.001)	0.01 (0.001)	1.6 (0.025)	0.9 (0.013)	< 0.1 (< 0.001)	0.8 (0.011)	< 0.2 (< 0.002)	1.7 (0.024)
Others	2.4 (0.039)	0.1 (0.002)	< 0.1 (< 0.001)	< 0.1 (< 0.001)	2.5 (0.041)	2.1 (0.031)	0.01 (0.002)	< 0.1 (< 0.002)	< 0.2 (< 0.002)	0.1 (0.002)
Total analysed	81.5 (1.352)	9.3 (0.154)	2.8 (0.046)	3.8 (0.063)	97.4 (1.615)	76.3 (1.119)	6.8 (0.100)	13.1 (0.192)	2.9 (0.043)	99.1 (1.454)
Unanalysed fraction				2.6 (0.043)	2.6 (0.043)				0.9 (0.013)	0.9 (0.013)
Total					100 (1.659)					100 (1.466)

Table 28 Proportion of radio-active components in different treated fractions of egg (pool of day 9–15) after administration of phenyl-^{14}C-cyclaniliprole

Compound	solvent	protease	acid	total
- parent	20.6 -0.143	< 0.2 (< 0.001)	0.4 -0.003	21 -0.146
- YT-1284	5.4 -0.038	0.4 -0.003	0.8 -0.005	6.6 -0.046
- NSY-28	53.1 -0.369	1 -0.007	0.3 -0.002	54.4 -0.378
- NSY-27	2.3 -0.016	< 0.2 (< 0.001)	< 0.1 (< 0.001)	2.3 -0.016
Hn-A (Rt~4-8 min)	0.2 -0.001	4.9 -0.034	0.1 -0.001	5.2 -0.036
Hn-B (Rt~9-11 min)	< 0.2 (< 0.001)	< 0.2 (< 0.001)	0.1 -0.001	0.1 -0.001
Hn-C (Rt~11-13 min)	< 0.2 (< 0.001)	< 0.2 (< 0.001)	< 0.1 (< 0.001)	< 0.5 (< 0.003)
Hn-D (Rt~14-16 min)	0.3 -0.002	< 0.02 (< 0.001)	0.2 -0.001	0.5 -0.003
Hn-E (Rt~17-19 min)	4.1 -0.028	0.4 -0.003	0.7 -0.005	5.2 -0.036
Hn-I (Rt~29-30 min)	2.7 -0.019	< 0.2 (< 0.001)	0.02 -0.001	2.9 -0.02
Others	< 0.2 (< 0.001)	0.2 -0.001	< 0.1 (< 0.001)	0.2 -0.001
Total analysed	88.7 -0.616	6.9 -0.048	2.8 -0.019	98.4 -0.683
Un-analysed hexane fraction				1.7 -0.012

Compound	solvent	protease	acid	total
Total				100 -0.695

Overview of the metabolic pathway of cyclaniliprole in livestock

Metabolism studies conducted with ruminants (lactating goats) and poultry (laying hens) and based on oral dosing, show that the metabolic pathways in ruminants and poultry are similar. The proposed biotransformation pathway for cyclaniliprole in the goat and hens is shown in Figure 3.

Metabolism observed arose via N-de-alkylation of cyclaniliprole and loss of 1-cyclopropylethyl group on the nitrogen atom in the amide moiety in the side chain yielding the corresponding primary amide, YT-1284. Subsequent cyclization by reaction between two amide moieties in YT-1284 gives the quinazoline compound, NSY-28 (pathway 1). YT-1284 can also be subjected to hydrolysis of the primary amide to give carboxylic acid NSY-27 (pathway 2).

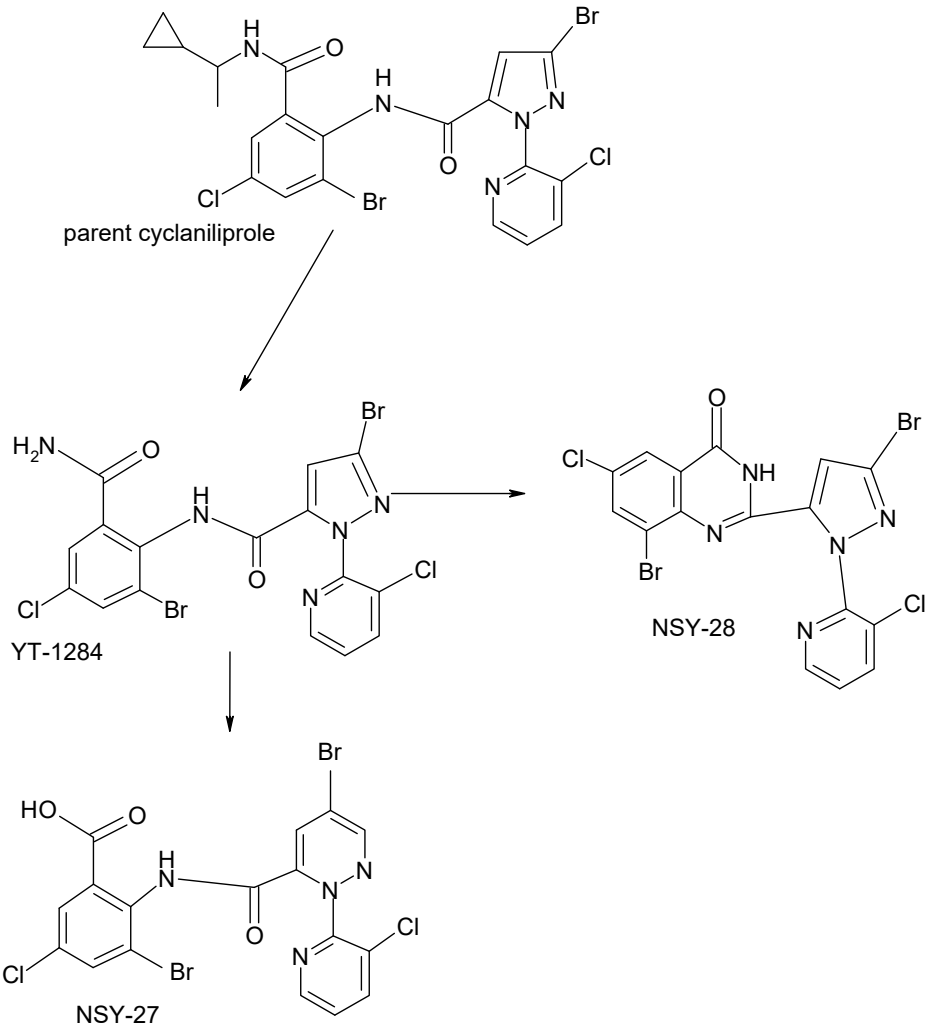


Figure 3 Metabolic pathway of cyclaniliprole in livestock

Environmental fate in soil

The Meeting received information on environmental fate in soil; a soil photolysis study [Button, 2011, report JSM0063], and aerobic soil degradation studies [Connor, 2013, report 13510.6102, McLaughlin, 2013, report 13510.6103]. According to the latest FAO manual (FAO paper 225, 2016, third edition[1], pp 37–38) none of these studies are required for foliar treatments.

Aerobic metabolism and degradation is soil

The route and rate of degradation of [^{14}C-Ph]-cyclaniliprole and [^{14}C-Pz]-cyclaniliprole were studied in a sandy clay loam soil in darkness under aerobic conditions in the laboratory at 20 °C and a moisture content of pF 2, over a period of 365 days [Connor, 2013, report 13510.6102]. Both biologically viable and sterile soils were studied.

The test system consisted of 250 mL glass incubation vessels containing 50 g (dry weight) soil attached to traps for the collection of CO_2 and volatile organics. Systems were incubated in darkness under aerobic conditions at a temperature of 20 ± 2 °C and maintained at a moisture level of pF 2.0.

Additional test vessels were established as sterile control samples for each of the radiolabels.

The application rate of the test substance was 0.2 mg/kg, equivalent to a field rate of 150 g ai/ha.

Soil samples were extracted and analysed immediately after sampling. Samples were extracted by shaking with acetonitrile : water (80:20 v:v). Extracts were separated from soil solids by centrifugation and aliquots were taken for LSC. The extraction procedure was repeated with acetonitrile : water : conc. HCl (80:20:0.1 v:v:v) and then acetonitrile : water : conc. HCl (80:20:0.5 v:v:v). Radioactivity in the soil solids following extraction was determined by combustion/LSC.

Extracts containing >3% of the applied radioactivity were combined. A portion of the combined extract was concentrated prior to reverse phase HPLC with radio-detection to quantify cyclaniliprole and its degradation products. The identity of the test substance in samples was established by co-chromatography with authentic cyclaniliprole.

The overall recovery of applied radioactivity (AR) from all non-sterile samples was in the range 94.7–100.9%. The distribution of radioactivity in each sample (extractedable, un-extracted and volatile) is shown in Table 29. Extracted radioactivity decreased from 96.6–97.6% AR at Day 0 to 66.6–68.9% AR at Day 365. Un-extracted radioactivity after the organic solvent extractions increased from 0.9–1.3% AR at Day 0 to 28.8–30.0% AR at Day 365, in sterile soil 19.6–2.6% AR at day 365. Volatile radioactivity as $^{14}CO_2$ accounted for a cumulative 0.6–1.0% AR after 365 days in non-sterile as well as sterile soil. There was negligible (≤ 0.1% AR) production of radioactivity as volatile organic compounds. The overall recovery of applied radioactivity (AR) from sterile samples was in the range 95.9–107.4%.

Table 29 Distribution and recovery of radioactivity from non-sterile and sterile aerobic soil treated with [^{14}C]-cyclaniliprole

Day	Extractable	NER	CO2[#]	VOC[#]	Total
	%AR	%AR	%AR	%AR	%AR
[^{14}C-Ph]-cyclaniliprole					
0	96.6	1.3	na	na	97.9
3	92.5	7.6	0.1	nd	100.3
31	81.0	14.8	0.2	nd	96.0
60	74.5	21.7	0.3	nd	96.5
122	76.8	21.3	0.3	0.1	98.5
180	73.2[a]	27.1	0.4	0.1	100.9
270	68.4[b]	26.8	0.5	0.1	95.8
365	66.6[c]	28.8	0.6	0.1	96.0
[^{14}C-Pz]-cyclaniliprole					

[1] http://www.fao.org/fileadmin/templates/agphome/documents/Pests_Pesticides/JMPR/Manual/FAO_manual_3rd_edition_Final.pdf

Day	Extractable	NER	CO_2[#]	VOC[#]	Total
	%AR	%AR	%AR	%AR	%AR
0	97.6	0.9	na	na	98.5
3	90.6	7.4	0.2	nd	98.2
31	81.7	14.3	0.4	nd	96.4
60	75.9	18.4	0.5	nd	94.7
122	75.3	22.8	0.6	nd	98.8
180	72.5[d]	23.5	0.7	nd	96.7
270	70.5[e]	27.4	1.0	nd	98.9
365	68.9[f]	30.0	1.0	nd	99.9
[^{14}C-Ph]-cyclaniliprole sterile					
0	95.6	1.1	na	na	96.7
120	87.9	7.8	0.1	nd	95.9
365	78.9	19.6	0.6	nd	99.1
[^{14}C-Pz]-cyclaniliprole sterile					
0	98.1	0.9	na	na	99.1
120	95.9	6.4	0.2	nd	102.5
365	83.9	22.6	0.9	nd	107.4

[#] presented cumulative; na: not applicable; nd: not detected

[a] Consisting of 71.5% parent and 1.7% NSY-27

[b] Consisting of 67.0% parent and 1.4% others

[c] Consisting of 63.3% parent, 0.8% NSY-27 and 2.4% others

[d] Consisting of 71.4% parent and 1.0% NSY-27

[e] Consisting of 96.3% parent and 1.2% others

[f] Consisting of 67.2% parent, 1.0% NSY-27 and 0.8% others

The distribution of cyclaniliprole and degradation products in the extractable soil fraction (non-sterile soils) is indicated with footnotes in Table 29. Metabolite NSY-27 was not detected in the sterile samples. In the non-sterile soil, cyclaniliprole was the major component at all times. Cyclaniliprole declined from 96.6–97.6% AR at Day 0 to 63.3–67.2% AR at Day 365. One minor product (NSY-27) was present at a maximum of 1.7% AR (at Day 180). In sterile soil, cyclaniliprole accounted for 78.9–83.9% AR at Day 365. The metabolite NSY-27 was not detected in sterile samples.

The DT_{50} and DT_{90} for the degradation of cyclaniliprole in non-sterile soil were calculated using the program CAKE (version 1.4, TESSELA) and reported to be DT_{50} of 444.6 days and DT_{90} of 1477 days. However, the residuals of the SFO fit show a clear pattern with time. A better fit, visually and statistically, can be obtained using DFOP. The calculated $DegT_{50}$ and $DegT_{90}$ for the degradation of cyclaniliprole were 692 days and 2300 days, respectively, see below.

model	g param	χ^2error%	DegT50 (d)	DegT90 (d)
SFO	na	5.06	692	2300
DFOP	0.17	1.41	1260[#]	

$DegT_{50}$ of the slow phase

Aerobic degradation in soil

The rate of degradation of cyclaniliprole was determined in four soils using (separately) [^{14}C-Ph]-cyclaniliprole and [^{14}C-Pz]-cyclaniliprole [McLaughlin, 2013, report 13510.6103]. The soil characteristics are summarised in Table 30.

Table 30 Characteristics of the test soils

Parameter	K Kenslow	S Spanish	M Marietta	L MSL
Origin	Derbyshire, UK	Valencia, Spain	Mississippi, USA	North Dakota, USA
Date of sampling from field	7 January 2013	6 November 2012	9 January 2013	19 December 2012
Particle size distribution (USDA classification):				
Sand (%)	55	43	59	61
Silt (%)	41	23	21	17
Clay (%)	4	34	20	22
Textural classification	Sandy loam	Clay loam	Sandy loam	Sandy clay loam
Bulk density, disturbed (g/cm^3)	0.96	1.12	1.09	1.06
Organic matter (%)	6.6	1.9	1.1	3.5
Organic carbon (%)[a]	3.9	1.1	0.65	2.1
pH (1:1 soil/water ratio)	5.2	7.8	7.9	6.8
Cation exchange capacity (meq/100 g)	10.4	17.0	12.6	17.1
Soil moisture:				
Capacity at 1/10 bar (pF 2.0) (%)	32.6	23.1	17.1	28.2
MWHC (g/100 g)	42.3	34.6	32.1	41.2
Microbial biomass-C (mg/100 g):				
Start	70.6	14.7	5.2	44.4
Middle	59.6	15.5	11.3	34.7
End	61.6	31.3	14.0	13.6

[a] % organic matter / 1.7

Cyclaniliprole was applied at a nominal rate of 0.2 µg/g, equivalent to a field rate of 150 g ai/ha.

The test system consisted of 250 mL glass incubation vessels containing 50 g (dry weight) soil attached to traps for the collection of CO_2 and volatile organics. Systems were incubated in darkness under aerobic conditions at a temperature of either 20 ± 2 °C or 35 ± 2 °C and maintained at a moisture level of pF 2.0. The microbial biomass of each soil was determined by the fumigation/extraction method prior to test initiation, during testing and at the end of the test.

Soil samples were processed (extracted) and analysed immediately after sampling. Samples incubated at 20 °C were initially extracted with 0.01 M $CaCl_2$ solution by shaking vigorously for 30 seconds followed by shaking at 150 rpm overnight. The samples were centrifuged and then extracted with acetonitrile : water (80:20 v:v), with acetonitrile : water : conc. HCl (80:20:0.1 v:v:v) and then acetonitrile : water : conc. HCl (80:20:0.5 v:v:v). All extracts were analysed by LSC. Radioactivity in the soil solids following extraction was determined by combustion/LSC. Extracts containing >3% of the applied radioactivity were combined, concentrated and analysed with reverse phase HPLC with radio-detection to quantify cyclaniliprole and its degradation products.

The results are shown in Table 31 and Table 32. Volatile organic compounds (VOCs) were not detected.

Table 31 Characteristics of residue in soils treated with cyclaniliprole (20 °C)

Kenslow								
Duration (days)	0	2	14	31	58	89	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$	4.5/4.6	2.5/2.4	4.1/3.7	2.7/2.5	2.2/2.2	1.5/1.7	2.2/2.1	2.3/2.4
Extracted	90.9/90.1	92.7/92.8	93.1/91.7	91.9/90.9	96.3/93.9	89.7/87.2	98.6/94.9	86.8/86.4
Un-extracted	0.6/1.2	1.3/1.8	2.7/2.8	2.3/2.0	3.6/2.5	4.2/3.4	4.4/3.7	5.4/5.2
CO_2	na/na	na/na	0.1/0.4	0.3/0.5	0.3/0.6	0.4/0.7	0.5/0.8	0.6/08
Total [%AR]	96.0/95.9	96.5/96.9	100.0/98.5	97.3/95.9	102.4/99.2	95.9/93.0	105.7/101.4	95.1/94.8
Spanish								
Duration (days)	0	2	14	31	58	89	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$	14.9/15.2	9.9/9.8	12.2/11.5	9.7/8.8	9.3/9.4	6.2/7.0	8.2/8.2	6.1/7.5
Extracted	78.5/78.5	84.9/84.6	82.5/83.6	84.5/85.3	86.4/86.7	84.9/83.8	89.8/88.8	78.8/80.2
Un-extracted	1.5/1.2	1.7/1.6	2.5/1.9	2.4/1.9	3.3/2.5	5.2/4.1	6.1/4.2	8.1/5.9

Kenslow								
Duration (days)	0	2	14	31	58	89	119	280
CO_2	na/na	na/na	0.2/0.3	0.3/0.3	0.5/0.4	0.6/0.5	0.8/0.5	0.8/0.6
Total [%AR]	94.8/94.9	96.4/96.0	97.4/97.3	96.9/96.4	99.5/99.0	96.9/95.4	104.8/101.7	93.8/94.2
Marietta soil								
Duration (days)	0	2	14	31	58	89	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$	27.6/27.8	16.0/15.5	19.6/19.7	16.3/15.9	14.4/15.2	8.9/10.5	12.3/12.1	9.3/10.0
Extracted	67.7/69.1	80.7/78.5	77.9/73.2	77.9/78.4	78.3/80.6	80.4/80.4	82.7/81.9	74.3/74.1
Un-extracted	1.3/0.7	1.4/1.2	2.1/3.0	1.5/1.8	3.2/2.7	5.4/4.2	5.8/5.7	8.8/8.5
CO_2	na/na	na/na	0.3/0.3	0.4/0.4	0.5/0.6	0.7/0.8	0.9/1.0	1.1/1.2
Total [%AR]	96.5/97.6	98.1/95.3	99.9/96.2	96.0/96.5	96.4/99.1	95.4/95.9	101.7/100.7	93.4/93.8
MSL								
Duration (days)	0	2	14	31	58	89	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$	7.4/8.1	4.8/5.4	6.7/7.4	3.9/3.8	3.0/3.9	2.4/3.5	2.9/3.7	2.1/2.8
Extracted	86.5/89.3	88.4/87.1	85.4/82.4	90.2/87.8	95.1/90.3	85.2/86.4	94.1/92.8	82.5/84.0
Un-extracted	1.3/1.0	1.9/1.3	2.5/3.4	2.4/3.2	2.1/2.2	5.3/4.2	5.0/4.5	6.9/5.7
CO_2	na/na	na/na	0.2/0.1	0.2/0.2	0.2/0.2	0.3/0.2	0.4/0.3	0.8/0.4
Total [%AR]	95.2/98.4	95.1/93.8	94.8/93.3	96.7/94.9	100.4/96.6	93.2/94.4	102.4/101.4	92.3/92.9

Table 32 Characteristics of residue in soils treated with cyclaniliprole (35 °C)

Kenslow							
Duration (days)	0	14	31	59	91	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$							
Extracted	96.5/100.3	92.4/93.7	91.7/92.3	93.1/94.8	89.6/89.0	86.8/89.1	82.9/89.7
Un-extracted	1.0/1.0	3.6/2.9	3.9/3.1	4.6/3.6	7.0/5.0	7.7/4.9	9.9/7.8
CO_2	na/na	0.2/0.4	0.3/0.4	0.3/0.5	0.4/0.5	0.6/0.6	0.7/0.7
Total [%AR]	97.5/101.3	96.1/96.9	95.9/95.8	98.1/98.9	97.0/94.5	95.1/94.6	93.5/98.1
Spanish							
Duration (days)	0	14	31	59	91	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$							
Extracted	98.3/97.6	89.8/91.1	89.5/89.2	87.1/92.2	85.5/86.7	86.6/87.2	79.9/79.0
Un-extracted	0.6/0.6	5.9/3.7	5.1/4.3	7.3/5.6	11.4/7.6	11.7/9.1	16.0/12.9
CO_2	na/na	0.1/0.2	0.3/0.4	0.4/0.5	0.4/0.6	0.5/0.6	0.7/0.7
Total [%AR]	98.9/98.1	95.8/95.0	94.9/94.0	94.7/98.2	97.3/94.8	98.8/97.0	96.5/92.6
Marietta soil							
Duration (days)	0	14	31	59	91	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$							
Extracted	100.0/100.3	89.8/90.7	89.4/88.9	88.3/91.6	81.5/85.9	78.7/84.9	76.9/76.9
Un-extracted	0.5/0.5	4.8/3.9	4.1/4.5	7.0/5.6	11.2/8.6	14.5/10.8	20.7/15.7
CO2	na/na	0.3/0.5	0.4/0.6	0.6/0.9	0.7/1.2	0.8/1.6	1.0/1.7
Total [%AR]	100.5/100.9	94.8/95.1	94.0/94.1	95.9/98.1	93.5/95.4	94.0/97.3	98.5/94.4
MSL							
Duration (days)	0	14	31	59	91	119	280
Characterisation	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR	%TAR
$CaCl_2$							
Extracted	100.7/100.3	90.5/90.9	69.8/89.3	92.5/94.0	87.8/88.4	86.2/87.6	81.6/85.2
Un-extracted	0.6/0.6	4.3/5.0	3.6/3.3	4.4/3.1	6.7/5.6	7.9/5.7	8.6/7.2
CO_2	na/na	0.2/0.4	0.3/0.6	0.5/0.8	0.5/0.9	0.6/0.9	0.6/1.4
Total [%AR]	101.3/100.9	95.1/96.4	73.7/93.1	97.4/97.8	95.0/94.9	94.7/94.3	90.9/93.8

The distribution of cyclaniliprole and degradation products in extracts is shown in Table 33 and Table 34. At 20 °C, the maximum amount of any individual metabolite was 2.6% AR. Two metabolites were identified as NSY-27 (maximum: 1.4%) and YT-1284 (maximum: 2.6%) on the basis of retention times observed for the standards.

Table 33 Distribution of cyclaniliprole and radiolabelled degradation products in the $CaCl_2$ extractable and organic extractable fractions of soil treated with [^{14}C]-cyclaniliprole and incubated at 20 °C, as % applied radioactivity

	label	Day	parent		YT-1284		NSY-27		Others	
			$CaCl_2$	Org.	$CaCl_2$	Org.	$CaCl_2$	Org.	$CaCl_2$	Org.
Kenslow	[^{14}C-Ph]	280	2.3	81.4	nd	nd	nd	1.39	nd	nd
Kenslow	[^{14}C-Pz]	31	2.5	88.5	nd	1.2	nd	0.4	nd	0.7
Kenslow	[^{14}C-Pz]	280	2.4	80.9	nd	1.9	nd	nd	nd	3.6
Spanish	[^{14}C-Ph]	31	9.7	83.5	nd	0.6	nd	0.4	nd	nd
Spanish	[^{14}C-Ph]	280	6.1	75.3	nd	nd	nd	nd	nd	3.5
Spanish	[^{14}C-Pz]	31	8.8	85.0	nd	nd	nd	nd	nd	0.3
Spanish	[^{14}C-Pz]	280	7.5	76.8	nd	nd	nd	nd	nd	3.4
Marietta	[^{14}C-Ph]	31	16.3	76.3	nd	0.4	nd	0.6	nd	0.6
Marietta	[^{14}C-Ph]	280	9.3	68.7	nd	nd	nd	nd	nd	5.6
Marietta	[^{14}C-Pz]	280	10.0	70.7	nd	nd	nd	nd	nd	3.4
MSL	[^{14}C-Ph]	31	3.9	87.9	nd	2.2	nd	nd	nd	nd
MSL	[^{14}C-Ph]	280	2.1	74.6	nd	2.6	nd	nd	nd	5.4
MSL	[^{14}C-Pz]	280	2.8	79.4	nd	1.4	nd	nd	nd	3.1

nd: not detected

Table 34 Distribution of cyclaniliprole and radiolabelled degradation products in the organic extractable fraction of soils treated with [^{14}C]-cyclaniliprole and incubated at 35 °C, as % applied radioactivity

Soil	Label	Day	parent	YT-1284	21.3 min[a]	Others
Kenslow	[^{14}C-Ph]	31	90.0	nd	nd	1.7
Kenslow	[^{14}C-Ph]	258	73.8	1.1	3.9	nd
Kenslow	[^{14}C-Pz]	258	73.3	1.8	4.9	5.9
Spanish	[^{14}C-Ph]	31	88.8	nd	nd	0.7
Spanish	[^{14}C-Ph]	258	72.4	1.2	2.9	nd
Spanish	[^{14}C-Pz]	258	64.2	nd	3.5	7.4
Marietta	[^{14}C-Pz]	258	70.3	nd	2.0	3.0
MSL	[^{14}C-Ph]	31	67.9	0.6	1.3	nd
MSL	[^{14}C-Ph]	258	72.2	0.6	3.4	nd
MSL	[^{14}C-Pz]	258	68.6	1.1	6.0	5.4

[a] Degradation product eluting at this time in the chromatographic system

nd: not detected

Extractedable cyclaniliprole as a function of time is stated in Table 33 and Table 34. DT_{50} and DT_{90} values for the degradation of cyclaniliprole were calculated using the program CAKE (version 1.4, TESSELA). Using data up to 280 days (20 °C) or 258 days (35 °C), both replicate values at each sampling time were used (not averaged) in a single kinetic evaluation. Single first order kinetics were assumed in a non-linear curve-fitting exercise. The calculated DT_{50} and DT_{90} values are shown in Table 35.

Table 35 Calculated $DegT_{50}$ and $DegT_{90}$ values for the degradation of cyclaniliprole

Soil	Incubation at 20 °C			Incubation at 35 °C		
	DT50 [days]	DT_{90} [days]	Chi^2 error	DT_{50} [days]	DT_{90} [days]	Chi^2 error
Kenslow	1118	3715	3.145	638	2119	2.021
Spanish	1048	3481	3.221	588	1953	2.386
Marietta	835	2775	2.534	548	1820	2.348
MSL	851	2828	4.081	482	1602	6.107

Remarks of the reviewer

The author calculated degradation constants for the 20 °C incubations assuming an additional data point at Day 0 with 100% cyclaniliprole. Consequently, all other data points were assumed to be one day later. This is considered incorrect. Half-lives were recalculated using CAKE version 3.2, see Table 36.

Table 36 Re-calculated $DegT_{50}$ and $DegT_{90}$ values for the degradation of cyclaniliprole

Soil	T (°C)	model	χ^2error%	$DegT_{50}$ (d)	$DegT_{90}$ (d)	$DegT_{50}$ (d) recalc.[a]
Kenslow	20	SFO	2.72	1710	5680	
Kenslow	35	SFO	2.02	638	2120	2360
Spanish	20	SFO	2.37	1730	5740	
Spanish	35	SFO	2.39	588	1950	2170
Marietta	20	SFO	1.74	1140	3780	
Marietta	35	SFO	2.35	548	1820	2020
MSL	20	SFO	3.14	1410	4680	
MSL	35	SFO	2.84	592	1970	2190

[a] Recalculated to reference conditions (T = 20 °C) using the default Eact of 65.4 kJ/mol (EFSA, 2007)

It is possible to calculate a substance specific Eact from the data, but the temperature of 35 °C is outside the range considered acceptable by EFSA. An average value of 46 kJ/mol was calculated, substantially lower than the default value.

Other degradation kinetics (DFOP, HS, and FOMC) were checked for better fits, but in all cases SFO fitted the data best.

In summary, degradation data were available for cyclaniliprole (5 trials at 20 °C) in soil under aerobic conditions. Four soils were also incubated at 35 °C but not further considered. In all cases, the derived DT_{50} value is longer than the incubation time of the experiment. In the original reports, these data were modelled using either simple graphical assessment or first-order methods. These data [MacLaughlin, 2013, report 13510.6103] were remodelled using CAKE version 3.2. For each dataset a statistical assessment was performed to establish the most appropriate model to fit to the data: simple first order (SFO) in study 02 [MacLaughlin, 2013, report 1510.6103] and double first order in parallel (DFOP) in study 01 [Connor, S, 2013, report 13510.6102]. Remodelled half-life values for cyclaniliprole ranged from 1140–1730 days (see Table 37). The median laboratory half-life determined for cyclaniliprole was 1410 days and the geometric mean was 1430 days (n=5).

Table 37 Trigger endpoint for cyclaniliprole for aerobic laboratory soil degradation studies

Soil name	Soil type	kinetic model (best fit)	χ^2error%	$DegT_{50}$ (days)	$DegT_{90}$ (days)	Ref, Report no
Kenslow	sandy loam	SFO	2.72	1710	5680	McLaughlin, 2013, report 13510.6103
Spanish	clay loam	SFO	2.37	1730	5740	idem
Marietta	sandy loam	SFO	1.74	1140	3780	idem
MSL	sandy clay loam	SFO	3.14	1410	4680	idem
Grand Folks	sandy clay loam	DFOP	1.41	1260[a]	not calculated	Connor, 2013, report 13510.6102

[a] Slow phase value: DT50 and DT 90 using with SFO 695 and 2300 days, respectively.

The proposed degradation pathway for cyclaniliprole under aerobic conditions is presented in Figure 4.

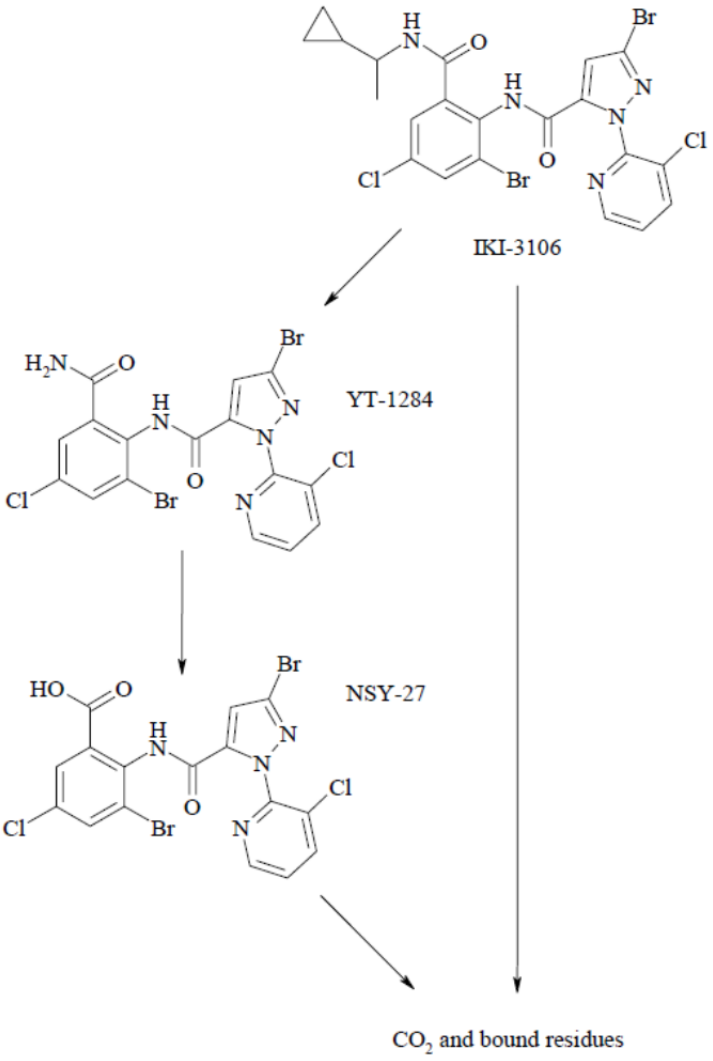


Figure 4 Proposed degradation pathways for cyclaniliprole in aerobic soil

Environmental fate in water/sediment systems

The Meeting did not receive information on the environmental fate in water/sediment systems. Studies on the environmental fate in water/sediments are not required for the envisaged uses.

RESIDUE ANALYSIS

The Meeting received information on enforcement/monitoring methods for the determination of cyclaniliprole and metabolites in plant and animal commodities. In addition, the Meeting received information on analytical methods for the determination of cyclaniliprole and metabolites as used in the various study reports (supervised residue trials, storage stability studies, processing studies, feeding studies).

The analytical residue methods have been evaluated according to the guidance provided by OECD (Series on Pesticides number 39) as indicated on page 29 of the FAO manual 2016.

Methods of analysis

For all analytical methods described below both cyclaniliprole and its metabolite NK-1375 are expressed as themselves, unless indicated differently. To express NK-1375 in cyclaniliprole equivalents a conversion factor of 1.064 can be applied.

Analytical methods for enforcement in animal commodities

LC-MS/MS Method JSM0277 [Airs, 2013, report JSM0277] was submitted as an analytical method for enforcement/monitoring method for the determination of cyclaniliprole and 4 relevant metabolites in animal commodities. The method is described below was also used in the animal feeding study [Ross, 2013, report JSM0515].

LC-MS/MS method JSM0277 for animal matrices

The general analytical method used in studies with animal commodities is described in Appendix 3 of report JSM0277 [Airs, 2013]. The procedure can be described as follows:

A sub-sample (2 g of tissue) is weighed into a 50 mL polypropylene tube and fortified if required. Acetonitrile (10 mL) is added, the sample homogenized, centrifuged and supernatant is transferred. Re-extraction of the solid sample with acetonitrile (10 mL) following procedures from first extraction. Supernatants are combined. Hexane is then added to the sample extract, shaken, centrifuged and phases separated. The hexane (upper) layer is discarded and the sample extract is diluted to 30 mL with acetonitrile. An aliquot (7.5 mL ≡ 0.5 g of matrix) of sample extract is transferred to a 50 mL graduated polypropylene tube and an aliquot (30 mL) of 0.2% acetic acid in water is added and subsequently mixed thoroughly.

For the SPE cleanup the Oasis HLB SPE cartridge is conditioned with methanol (5 mL) and water (5 mL), discarding the eluate. The extracted samples are loaded onto the SPE cartridge, discarding the eluate. The cartridge is washed with an aliquot (2 mL) of water, discarding the eluate, followed by airdrying of the cartridge. The SPE cartridge is subsequently eluted with an aliquot (6 mL) of methanol and the final extract diluted to 7.5 mL with methanol to a final matrix concentration ≡ 0.0667 g matrix / mL using further dilutions with methanol as required. Samples are then quantified by the use of LC-MS/MS.

LC-MS/MS (mobile phase A: water:methanol:formic acid (90:10:0.1 v/v/v) containing 0.01 M ammonium formate and mobile phase B: methanol: formic acid (100:0.01 v/v)). The ion transitions monitored were *m/z* 602→286 (cyclaniliprole), *m/z* 566→498 (NK-1375), *m/z* 535→284 (NSY-27), *m/z* 516→480 (NSY-28) and *m/z* 534→284 (YT-1284) which were monitored for quantitation purposes. To demonstrate possible confirmation of residues, alternative ion transitions, 604→286 (cyclaniliprole), *m/z* 568→500 (NK-1375), *m/z* 537→286 (NSY-27), *m/z* 518→482 (NSY-28) and *m/z* 536→286 (YT-1284) are monitored. LOQ of the method is 0.01 mg/kg for parent cyclaniliprole and for metabolites NK-1375, NSY-28, NSY 27, and YT-1284.

LC-MS/MS method JSM0277 was developed and validated according to the full validation scheme for the determination of cyclaniliprole and 4 metabolites (NK-1375, NSY-27, NSY-28 and YT-1284) in hen eggs and bovine liver, kidney, muscle, fat and milk [Airs, 2013, report JSM0277].

The validation was performed for the quantification transition ions as well as for the confirmation transition ions. Samples were fortified with known concentrations of cyclaniliprole, NK-1375, NSY-27, NSY-28 and YT-1284 standard before extraction. The reported LOQ was 0.01 mg/kg for each commodity and each analyte. The results are summarised in Table 38. Average recoveries at 0.01 mg/kg and 0.1 mg/kg of parent or metabolites were within 70–120% limits and RSDs were within 20%. Linearity of detector response (correlation coefficient $r > 0.99$) was observed in the range of 0.2–20 ng/mL for parent and the four metabolites. This range is equivalent to 0.3–30× LOQ (0.003 to 0.3 mg/kg in matrix) in the samples.

No significant (greater than 20%) enhancement or suppression of response was observed for the analytes in the final sample extracts for hen eggs, bovine kidney, muscle, fat and milk.

Significant enhancement (greater than 20%) was observed for bovine liver for one of the analytes (YT-1284). Therefore matrix-matched calibration standards were used for this matrix.

Control (untreated) samples of each of the six matrices were analysed and were below the LOQ of 0.01 mg/kg. There was no apparent response (i.e. < 30% of the LOQ) in the region of the

chromatograms corresponding to the retention time of cyclaniliprole, NK-1375, NSY-27, NSY-28 and YT-1284.

The results from this test demonstrate that the analytes are stable in the final extracts when stored at approximately -20 °C. The storage period tested was 7 days.

An independent laboratory validation of LC-MS/MS method JSM0277 for the determination of cyclaniliprole and metabolites in animal tissues (bovine liver, muscle, whole milk and hen egg) was performed [Schulz and Herrig, 2013, report S13-03806].

The animal tissues were extracted and analysed according to the procedures described in method JSM0277. In untreated samples of each matrix, there was no apparent response (i.e. < 30% of the LOQ) in the regions of the chromatogram at the retention times of cyclaniliprole, NK-1375, NSY-27, NSY-28 and YT-1284. The response of the system to standard solutions of the five analytes was linear over the range 0.2 to 20 ng/mL (equivalent to 0.003 to 0.3 mg/kg in the matrix) for both the quantification and confirmatory transitions ($r \geq 0.9988$ in data reported). Though some values were below 70% or above 120%, the average recoveries obtained were within guideline requirements. Precision was within requirements (RSD ≤ 20%). The LOQ, defined as the lowest fortification level at which acceptable recovery data were obtained, was 0.01 mg/kg for all five analytes in the six matrices tested. Control (untreated) samples of each of the six matrices were analysed and were below the reported <LOQ of 0.01 mg/kg.

Validation results are summarised in Table 38. The validation of the methodology for the determination of parent cyclaniliprole and metabolites NK-1375, NSY-27, NSY-28, and YT-1284 in the four matrix types demonstrated that they could be accurately determined at 0.01 and 0.1 mg/kg. The additional analysis of the validation samples using an alternative MS-MS ion transition (not reported in the table) for each analyte demonstrated a suitable confirmatory technique (cyclaniliprole *m/z* 602→177, NK-1375 *m/z* 566→266). A stability test showed that the analytes were stable in the final extracts when stored at approximately -20 °C for seven days.

Table 38 Validation results and concurrent method recoveries for LC-MS/MS method in animal commodities

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
parent cyclaniliprole						
bovine fat	0.01 0.1	90 88-92 91 83-99	2.3 8	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear $r \geq 0.9988$	JSM0277
bovine fat, sub-cutaneous	0.01 0.1	na 89 na 87	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear $r = 0.9999$	JSM0515
bovine fat, perirenal	0.01 0.1	na 66 na 81	na na	1 1	idem	JSM0515
bovine fat, omental	0.01 0.1	na 88 na 65	na na	1 1	idem	JSM0515
bovine kidney	0.01 0.1	102 97-105 104 95-109	3.4 5.2	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear $r \geq 0.9988$	JSM0277
bovine kidney	0.01 0.1	na 90 na 86	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear $r = 0.9999$	JSM0515
bovine liver	0.01	93 85-102	7.6	5	external standards	JSM0277

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
	0.1	101 94-108	6.2	5	0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	
bovine liver	0.01 0.1	na 99 na 94	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine liver	0.01 0.1	106 96-118 103 94-118	8.1 9.4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine muscle	0.01 0.1	81 74-90 86 80-91	7.4 5.5	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine muscle	0.01 0.1	105 91-119 95 91-99	9.6 6	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine muscle	0.01 0.1	na 96 na 87	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine milk	0.01 0.1	90 83-98 92 90-94	5.9 1.7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine whole milk	0.01 0.1	101 96-105 95 91-99	3.6 3.1	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine, whole milk	0.01 0.1	85 74-109 79 70-92	11.5 9.4	13 13	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine, skimmed milk	0.01 0.1	74 73, 74 73 70, 76	na na	2 2	idem	idem
bovine, cream	0.01 0.1	92 85, 100 76 70, 82	na na	2 2	idem	idem
hen egg	0.01 0.1	85 81-90 97 94-98	4.6 1.7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
hen egg	0.01 0.1	108 100-116 100 96-105	5.3 3.4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
metabolite NK-1375						
bovine fat	0.01 0.1	100 96-104 96 89-101	3 4.9	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear	JSM0277

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
					r ≥ 0.9988	
bovine fat, sub-cutaneous	0.01 0.1	na 92 na 88	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 1.0000	JSM0515
bovine fat, perirenal	0.01 0.1	na 78 na 85	na na	1 1	idem	JSM0515
bovine fat, omental	0.01 0.1	na 98 na 63 [a]	na na	1 1	idem	JSM0515
bovine kidney	0.01 0.1	85 84-86 82 74-86	1 5.7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine kidney	0.01 0.1	na 90 na 98	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 1.0000	JSM0515
bovine liver	0.01 0.1	91 82-100 94 87-101	7.4 6.2	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine liver	0.01 0.1	103 92-113 101 91-124	8.4 13	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine liver	0.01 0.1	na 95 na 92	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 1.0000	JSM0515
bovine muscle	0.01 0.1	82 76-87 76 72-79	5.1 3.6	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine muscle	0.01 0.1	94 86-107 85 79-89	8.6 4.2	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine muscle	0.01 0.1	na 101 na 88	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 1.0000	JSM0515
bovine milk	0.01 0.1	104 101-108 102 100-106	2.4 2.4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine whole milk	0.01 0.1	98 93-102 90 86-96	3.4 4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine, whole milk	0.01	78 68-99	11.7	13	external standards	JSM0515

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
	0.1	75 65-86	8.9	13	0.2 ng/mL to 20 ng/mL, linear r = 1.0000	
bovine, skimmed milk	0.01 0.1	74 73, 75 72 70, 73	na na	2 2	idem	idem
bovine, cream	0.01 0.1	80 70, 90 77 69, 85	na na	2 2	idem	idem
hen egg	0.01 0.1	92 89-97 93 89-97	3.4 3.1	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
hen egg	0.01 0.1	101 93-111 93 89-97	6.5 3.5	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
metabolite NSY-27						
bovine fat	0.01 0.1	100 92-108 93 89-95	6.4 4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine fat, sub-cutaneous	0.01 0.1	na 87 na 102	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9976	JSM0515
bovine fat, perirenal	0.01 0.1	na 65 [a] na 90	na na	1 1	idem	JSM0515
bovine fat, omental	0.01 0.1	na 82 na 63 [a]	na na	1 1	idem	JSM0515
bovine kidney	0.01 0.1	98 81-114 94 89-97	14.4 4.1	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine kidney	0.01 0.1	na 81 na 88	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9976	JSM0515
bovine liver	0.01 0.1	83 75-94 85 80-90	12.2 4.7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine liver	0.01 0.1	103 93-112 92 83-116	8.6 15	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine liver	0.01 0.1	na 83 na 91	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9976	JSM0515
bovine muscle	0.01 0.1	88 76-93 82 75-88	7.9 6.8	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
bovine muscle	0.01 0.1	103 87-118 90 85-93	12 3.8	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine muscle	0.01 0.1	na 96 na 83	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9976	JSM0515
bovine milk	0.01 0.1	94 89-110 90 86-95	11.4 4.1	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine whole milk	0.01 0.1	93 91-95 90 85-92	1.6 3.1	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine, whole milk	0.01 0.1	87 71-104 85 72-97	13.2 9.1	13 13	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9976	JSM0515
bovine, skimmed milk	0.01 0.1	74 74, 75 73 73, 73	na na	2 2	idem	idem
bovine, cream	0.01 0.1	88 74, 101 80 74, 87	na na	2 2	idem	idem
hen egg	0.01 0.1	95 75-104 91 88-95	12.3 2.9	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
hen egg	0.01 0.1	96 88-102 93 90-97	5.8 2.8	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
metabolite NSY-28						
bovine fat	0.01 0.1	95 81-110 84 72-95	14.6 10.7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine fat, sub-cutaneous	0.01 0.1	na 84 na 78	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9997	JSM0515
bovine fat, perirenal	0.01 0.1	na nr na 90	na na	1 1	idem	JSM0515
bovine fat, omental	0.01 0.1	na 93 na nr	na na	1 1	idem	JSM0515
bovine kidney	0.01 0.1	87 77-97 85 75-91	10.7 7.6	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine kidney	0.01 0.1	na 82 na 87	na na	1 1	external standards 0.2 ng/mL to 20	JSM0515

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
					ng/mL, linear r = 0.9997	
bovine liver	0.01 0.1	81 70-95 90 82-103	12.5 9	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine liver	0.01 0.1	72 65-89 76 67-98	15 16	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine liver	0.01 0.1	na 93 na 89	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9997	JSM0515
bovine muscle	0.01 0.1	93 78-98 86 82-89	9.1 3.6	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine muscle	0.01 0.1	84 70-102 79 73-84	15 5.6	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine muscle	0.01 0.1	na 104 na 70	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9997	JSM0515
bovine milk	0.01 0.1	84 70-101 82 79-88	13.6 4.4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine whole milk	0.01 0.1	81 78-84 74 72-76	3.2 3.2	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine, whole milk	0.01 0.1	79 71-96 73 63-80	8.2 6.2	13 12	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9997	JSM0515
bovine, skimmed milk	0.01 0.1	77 76, 78 70 70, 71	na na	2 2	idem	idem
bovine, cream	0.01 0.1	81 80, 82 74 73, 75	na na	2 2	idem	idem
hen egg	0.01 0.1	77 73-83 88 80-95	5.5 7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
hen egg	0.01 0.1	106 100-115 98 97-100	6 1.8	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
metabolite YT-1284						

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
bovine fat	0.01 0.1	106 96-113 103 90-115	6.3 8.9	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine fat, sub-cutaneous	0.01 0.1	na 106 na 102	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine fat, perirenal	0.01 0.1	na 91 na 101	na na	1 1	idem	JSM0515
bovine fat, omental	0.01 0.1	na 103 na 88	na na	1 1	idem	JSM0515
bovine kidney	0.01 0.1	105 94-110 101 95-105	6 4.3	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine kidney	0.01 0.1	na 77 na 94	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine liver	0.01 0.1	92 82-101 97 88-103	8.6 5.9	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine liver	0.01 0.1	108 103-116 104 92-129	5.1 14	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine liver	0.01 0.1	na 97 na 100	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine muscle	0.01 0.1	101 96-105 93 90-98	4.3 3.3	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine muscle	0.01 0.1	104 84-130 92 83-95	17 5.7	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)
bovine muscle	0.01 0.1	na 93 na 94	na na	1 1	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine milk	0.01 0.1	101 91-109 101 98-104	7 2.4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
bovine whole milk	0.01 0.1	98 93-102 97 93-99	3.5 2.6	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear	S13-03806 (ILV of method JSM0277)

Matrix	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	calibration	Code no; Report no
					r ≥ 0.9988	
bovine, whole milk	0.01 0.1	88 70-105 89 75-109	11.8 12.2	13 12	external standards 0.2 ng/mL to 20 ng/mL, linear r = 0.9999	JSM0515
bovine, skimmed milk	0.01 0.1	84 81, 88 88 87, 88	na na	2 2	idem	idem
bovine, cream	0.01 0.1	97 95, 99 84 79, 90	na na	2 2	idem	idem
hen egg	0.01 0.1	95 81-104 95 90-103	9.1 5.4	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	JSM0277
hen egg	0.01 0.1	98 92-106 94 92-97	5.5 2.2	5 5	external standards 0.2 ng/mL to 20 ng/mL, linear r ≥ 0.9988	S13-03806 (ILV of method JSM0277)

na=not applicable; nr = not reported

A radiovalidation study [Unsworth, 2014a, report JSM0493] was carried out to evaluate the efficiency of extraction and analysis of cyclaniliprole and its metabolites NK-1375, NSY-27, NSY-28, and YT-1284, using the residue analytical method JSM0277. Samples of animal tissues from goat (liver and milk) and hen (liver and eggs) metabolism studies were analysed using method JSM0277. Solvent extracts from this method were then analysed by the radioanalytical methods used on the metabolism studies [Kane, 2013 report JSM0059 and Jones, 2013, report JSM0060]. Results from the three methods were then compared. The extraction efficiency of the residue analytical method, ranging from 57.2% TRR in hen liver to 84.4%TRR in hen egg, was in agreement with the extraction efficiency of the method used in the metabolism studies (81.5–98.2 5TRR). The relative extraction efficiency of the residue analytical method to the extraction method from the metabolism studies ranged from 70.2–93.4%. The results are presented in Table 39.

Table 39 Comparison of the extraction efficiency of the residue analytical method with the extraction efficiency of the methods from the livestock metabolism studies

	Extraction efficiency [%]		
	Residue analytical method	Metabolism study methods[a]	Relative extraction efficiency
Goat liver	65.0	85.8	75.8
Goat milk	71.5	98.2	72.8
Hen liver	57.2	81.5	70.2
Hen egg	84.4	90.4	93.4

[a] Neutral solvent extracts (Studies JSM0059 and JSM0060, goat and hen respectively)

The concentrations of cyclaniliprole and its metabolites NSY-27, NSY-28, YT-1284, NK-1375 and NSY-137 are comparable with either LC-MS/MS, radio-HPLC or Radio-TLC. This confirmed that the LC-MS/MS method JSM0277 was suitable for the analysis of cyclaniliprole and its metabolites in these samples. Results are presented in Table 40.

Table 40 Mean residues of parent cyclaniliprole and its metabolites in animal tissues in mg/kg analyte

Compound	goat					
	liver			milk		
	LC-MS/MS	radio-HPLC	Radio-TLC	LC-MS/MS	radio-HPLC	Radio-TLC
Cyclaniliprole	0.40	0.32	0.35	0.08	0.08	0.08
NSY-27	< 0.01	0.01	0.02	nd	< 0.01	nd

Compound	goat					
	liver			milk		
	LC-MS/MS	radio-HPLC	Radio-TLC	LC-MS/MS	radio-HPLC	Radio-TLC
NSY-28	0.19	0.31	0.25	< 0.01	< 0.01	< 0.01
YT-1284	0.14	0.14	0.12	< 0.01	< 0.01	< 0.01
NK-1375	nd	nd	nd	nd	nd	nd
NSY-137	nd	nd	nd	nd	nd	nd
	hen					
	liver			Egg		
Cyclaniliprole	0.03	0.03	0.03	0.20	0.20	0.22
NSY-27	0.05	0.02	0.05	0.01	0.05	< 0.01
NSY-28	0.52	0.78	0.78	0.43	0.43	0.50
YT-1284	0.03	0.04	0.03	0.03	0.02	0.02
NK-1375	nd	nd	nd	nd	nd	nd
NSY-137	nd	0.02	nd	nd	nd	nd

nd = not detectable

Reviewer's conclusion: LC-MS/MS Method JSM0277 is considered:

- valid (full validation) for the determination of cyclaniliprole in hen eggs, bovine, fat, kidney, liver, muscle and milk (0.01–0.1 mg/kg).
- valid (full validation) for the determination of metabolites NK-1375, NSY27, YT-1284, NSY 28 in hen eggs, bovine, fat, kidney, liver, muscle and milk (0.01–0.1 mg/kg for each analyte).
- valid for the determination of cyclaniliprole and metabolites NK-1375, NSY27, YT-1284, NSY 28 (radiovalidation) in goat liver and milk, and hen egg and liver.

The valid LOQ is 0.01 mg/kg per analyte (no validations below this level).

Analytical methods for enforcement in plant commodities

Multi residue method QuEChERS (AOAC 2007.01) LC-MS/MS

The analytical methodology for the determination of residues of cyclaniliprole and NK-1375 in food of plant origin using the multi residue method QuEChERS (AOAC 2007.01) was validated in food of plant origin [Miller, 2015, report JSM0755]. Untreated samples of grape, peach, avocado and soya bean were used. Samples were cut, and frozen at -20 °C. Frozen samples were homogenized by grinding.

Samples of 15 gram (7.5 g for soy bean low water content) are weighed. 15 mL of acetonitrile with 1% acetic acid is added and shaken. Component of extraction kit, 6 g magnesium sulfate and 1.5 g sodium acetate is added. Followed by addition of 0.075 mL of 40 µg/mL (20 µg/mL for soya bean) isofetamid in acetonitrile as internal standard. After shaking and centrifuging supernatant is transferred into PTFE centrifuge tube, component of dispersive kit, 400 mg PSA and 1200 mg magnesium sulfate is added. After shaking and centrifuging, the supernatant is diluted with acetonitrile and analysed by LC-MS/MS (cyclaniliprole *m/z* 602>284, NK-1375 *m/z* 566>498). The recovery data using a second ion transition produced comparable result to the analysis of the quantitation ion transition (cyclaniliprole *m/z* 602>177, NK-1375 *m/z* 566>266). Linearity, specificity, accuracy and precision are presented in Table 41 and Table 42. Limit of detection was defined as the concentration of the lowest calibration standard chromatographed that gave rise to a measurable chromatographic response. The LOQ of the method was 0.01 mg/kg for both analytes. Matrix effects: No significant enhancement or suppression of response was observed. Both analytes were stable in the final extracts for 7 days at -20 °C in dark condition (96–107% recovery).

A second method validation for the determination of cyclaniliprole and its metabolite (NK-1375) in food of plant origin by QuEChERS [Nakano, 2014, report IRA14028G] was performed. A sample material (grape, peach, avocado, soya bean) was shaken with acetonitrile containing 1% acetic acid for 1 minute. After addition of a mixture of anhydrous magnesium sulfate and sodium acetate the

samples was shaken again and centrifuged. The extract was cleaned with PSA (primary secondary amino phase) and analysed with reversed phase HPLC with MS/MS detection (EXI, positive ion mode, with mass transition: cyclaniliprole *m/z* 602→284, NK-1375 *m/z* 566→498)). Quantification was performed with matrix matched calibration standards. Linearity, specificity, accuracy and precision are presented in Table 41 and Table 42. Limit of detection was defined as 30% of the LOQ of 0.01 mg/kg. Matrix effects: Significant suppression of response for cyclaniliprole, NK-1375 and internal standard (isofetamid) was observed by the final sample extract of avocados and slight suppression of response for each analyte at 0.01 mg/kg fortified was observed by the final samples extract of grapes, peaches and soya beans. Both analytes were stable in the final extracts for 7 days at -20 °C in dark condition (96–107% recovery).

Table 41 Validation results for detection of cyclaniliprole with QueEChERS method (AOAC 2007.01) in plant commodities

Commodity	Reported LOQ mg/kg	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r (%)	n	Control mg/kg	Calibration	Reference, method
avocado	0.01	0.01 0.1	89 101	87-90 100-103	1.7 1.5	5 5	< 0.01 (n=2)	cyclaniliprole and NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=0.9983	IRA14028G, QuEChERS (AOAC 2007.01) ILV
grape	0.01	0.01 0.5	98 92	94-102 88-96	3.1 4.1	5 5	< 0.01 (n=2)	standard solution of cyclaniliprole and NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9993	JSM0755 QuEChERS (AOAC 2007.01) ILV
grape	0.01	0.01 0.1	93 98	93-94 97-99	0.6 0.9	5 5	< 0.01 (n=2)	cyclaniliprole and NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=0.9987	IRA14028G, QuEChERS (AOAC 2007.01) ILV
peach	0.01	0.01 0.5	95 90	92-101 85-93	3.8 3.4	5 5	< 0.01 (n=2)	standard solution of cyclaniliprole and NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9993	JSM0755 QuEChERS (AOAC 2007.01) ILV
peach	0.01	0.01 0.1	99 108	98-101 105-111	1.2 2.1	5 5	< 0.01 (n=2)	cyclaniliprole and NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=1.000	IRA14028G, QuEChERS (AOAC 2007.01) ILV
oil seed rape	0.01	0.01 0.5	99 103	94-107 100-106	4.8 2.1	5 5	< 0.01 (n=2)	standard solution of cyclaniliprole and NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9993	JSM0755 QuEChERS (AOAC 2007.01) ILV
bean, dry	0.01	0.01 0.5	101 94	98-107 93-98	3.6 2.3	5 5	< 0.01 (n=2)	standard solution of cyclaniliprole and NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9993	JSM0755 QuEChERS (AOAC 2007.01) ILV
soybean	0.01	0.01 0.1	94 101	91-97 97-105	2.8 3.4	5 5	< 0.01 (n=2)	cyclaniliprole and NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=0.9989	IRA14028G, QuEChERS (AOAC 2007.01) ILV

Table 42 Validation results for detection metabolite NK-1375 QueEChERS method (AOAC 2007.01) in plant commodities

Commodity	Reported LOQ mg/kg	Spike level mg/kg	% Recovery mean	% Recovery range	RSD_r	n	Control mg/kg	Calibration	Reference, method
avocado	0.01		84	82-86	1.8%	5	< 0.01 (n=2)	standard solution of NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=0.9986	IRA14028G, QuEChERS (AOAC 2007.01) ILV idem
		0.1	91	90-91	0.5%	5			
grape	0.01		101	96-106	4.4%	5	< 0.01 (n=2)	standard NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9973	JSM0755 QuEChERS (AOAC 2007.01) ILV
		0.5	93	89-100	5.0%	5			
grape	0.01		93	92-93	1.3%	5	< 0.01 (n=2)	NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=0.9987	IRA14028G, QuEChERS (AOAC 2007.01) ILV
		0.1	97	97-98	0.6%	5			
peach	0.01	0.01	97	93-102	3.5%	5	< 0.01 (n=2)	standard solution of NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9973	JSM0755 QuEChERS (AOAC 2007.01) ILV
		0.5	91	85-95	4.3%	5			
peach	0.01		98	97-101	1.5%	5	< 0.01 (n=2)	NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=1.000	IRA14028G, QuEChERS (AOAC 2007.01) ILV
		0.1	104	102-106	1.7%	5			
oil seed rape	0.01		99	92-102	4.1%	5	< 0.01 (n=2)	standard solution of cyclaniliprole and NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9973	JSM0755 QuEChERS (AOAC 2007.01) ILV
		0.5	107	101-110	3.3%	5			
bean, dry	0.01		99	97-104	3.1%	5	< 0.01 (n=2)	standard solution of NK-1375 0.3-10 ng/mL and internal standard isofetamid linear, r≥0.9973	JSM0755 QuEChERS (AOAC 2007.01) ILV
		0.5	96	94-100	2.6%	5			
Soya bean	0.01		91	87-94	3.0%	5	< 0.01 (n=2)	NK-1375 0.3-100 ng/mL and internal standard isofetamid linear, r=0.9977	IRA14028G, QuEChERS (AOAC 2007.01) ILV
		0.1	98	94-99	2.2%	5			

Analytical methods used in study reports in plant commodities

LC-MS/MS Method JSM0269 for plant matrices

In the various studies in plant matrices (residue trials, metabolism studies and crop rotation studies) the same LC-MS/MS analytical method was applied. The method [Brewin, 2012, report JSM0269] determines parent compound cyclaniliprole and metabolite NK-1375 as two different analytes. Residues are expressed as their respective identity. The reported LOQ is 0.01 mg/kg for each analyte. A description of the method is described as follows:

Samples are extracted twice with acetonitrile. For matrices with low water content (e.g. dry beans), add an aliquot (20 mL) of water and allow to stand for approximately 20 minutes before extraction with acetonitrile. Supernatants are combined and diluted with acetonitrile. The extract is cleaned by solid-phase extraction (SPE) using the Oasis HLB SPE cartridge. The extract is loaded and the column washed with acetontrile:water (40:60 v:v) followed by drying of the cartridge. Then the cartridge is eluted with acetonitrile and the finale eluate is diluted to 50 mL with acetonitrile. Quantitation was performed using liquid chromatography with tandem mass spectrometric detection (LC-MS/MS).

The parent cyclaniliprole (IKI-3106) is quantified by GC-MS/MS at m/z 602 using an external standard. Cyclaniliprole: *mlz* 602→284; cyclaniliprole: *mlz* 602→ 177 (confirmatory). Metabolite NK-1375 is quantified by GC-MS/MS at *m/z* 566 using an external standard. NK-1375 at *mlz* 566→498; NK-1375: *m/z* 566→266 (confirmatory).

The method was used in apples [Schäufele, 2013a, report JSM0347; Schäufele, 2013b, report JSM0348; Schäufele, 2013c, report JSM0473; Schäufele, 2016a, report SQ74KP (processing); Wiedmann and McDonald, 2013b, report IB-2012-JLW-020, Wiedmann and McDonald, 2014a, report IB-2013-JLW-004; Farrell, 2013a, report ULP-1113; Farrell, 2013b, report ISK12433; Miller, 2016a, XR44SB] and pears [Wiedmann and McDonald, 2014a, report IB-2013-JLW-004], cherries [Wiedmann and McDonald, 2013c, report IB-2012-JLW-005], plums [Schäufele, 2013d, report JSM0338; Schäufele, 2013e, report JSM0476; Wiedmann and McDonald, 2013c, report IB-2012-JLW-005], prunes [Wiedmann and McDonald, 2013c, report IB-2012-JLW-005], apricots [Schäufele, 2013f, report JSM0329; Schäufele, 2013g, report JSM0474; Schäufele, 2014a, report JSM0667], peaches [Schäufele, 2013h, report JSM0351; Schäufele, 2013i, report JSM0352; Schäufele, 2013j, report JSM0475, Wiedmann and McDonald, 2013c, report IB-2012-JLW-005; Schaufele, 2016b, report QK27SS (processing)], wine and table grapes [Schäufele, 2013k, report JSM0330; Schäufele, 2013l, report JSM0477; Schäufele, 2013m, report JSM0349; Schäufele, 2013n, report JSM0350; Schäufele, 2013o, report JSM0478; McDonald, 2014a, report IB-JAM-002], broccoli [Alé, 2013a, report JSM0333; Alé, 2013b, report JSM0481; Wiedmann and MacDonald, 2014b IB-2012-JLW-28], Brussels sprouts [Alé, 2013c, report JSM0340, Alé, 2013d, report JSM0484, Alé 2014, report JSM0603], cauliflower [Alé, 2013e, report JSM0332; Alé, 2013f, report JSM0480; Wiedmann and MacDonald, 2014b IB-2012-JLW-28], head cabbages [Alé, 2013g, report JSM0334; Alé, 2013h, report JSM0482; Wiedmann and MacDonald, 2014b IB-2012-JLW-028], cucumbers, summer squash and cantaloupe [McDonald and Wiedmann, 2014b, report IB-2013-JAM-003-01], sweet pepper outdoor [Alé, 2013i, report JSM0336; Alé, 2013j, report JSM0485; Wiedmann, 2014c, IB2012JLW029-0101] and sweet pepper indoor [Alé, 2013k, report JSM0337; Alé, 2013l, report JSM0487] and tomato outdoor [Alé, 2013m, report JSM0335; Alé, 2013n, report JSM0486; Wiedmann, 2014c, IB2012JLW0290101; Schäufele, 2016c, report HH97BD (processing)] and tomato indoor [Alé, 2013o, report JSM0353; Alé, 2013p, report JSM0354; Alé, 2013q, report JSM0488], kale [Alé, 2013r, JSM0483], mustard greens [Wiedmann and MacDonald, 2014b IB-2012-JLW-028], lettuce and spinage [MacDonald and Wiedmann, 2013c, IB-2012-JAM-001-01-01], tree nuts (almond and pecan) [Wiedmann and McDonald, 2014d, report IB-2012-JLW-019-01-01], wheat, forage, hay, grain and straw [Bartolomé, 2013, report JSM0414, Wiedmann and McDonald, 2013a, report IB-2012-JLW-022-01-01]. Procedural recoveries and method validations are summarised in Table 45 and Table 46.

A validation study for this method is available in the same report [Brewin, 2012, report JSM0269]. In this study the method was validated for the determination of cyclaniliprole and its metabolite NK-1375 in five matrix types (grape, wine, peaches, oilseed rape seeds and dry beans). Samples with low water content were allowed to stand for 20 minutes with water added prior to extraction. Samples were extracted with acetonitrile. Matrices with low water content and oily matrices were homogenised for approximately 30 seconds. After shaking, centrifugation, the supernatant was decanted and the solid sample residue was again extracted with acetonitrile by shaking by hand. Supernatant were combined and diluted to 200 mL with acetonitrile, prior to solid phase extraction (SPE) cleanup. Quantitation was performed using liquid chromatography with

tandem mass spectrometric detection (LC-MS/MS). Quantitation ion transition: cyclaniliprole *m/z* 602→284, NK-1375 *m/z* 566→498.

The method was validated at 0.01 and 0.05 mg/kg for quantification of both analytes in the matrix samples tested. Validation results are summarised in Table 45 and Table 46. The validation of the methodology for the determination of cylaniliprole or NK-1375 in the five matrix types demonstrated that they could be accurately determined at 0.01 and 0.5 mg/kg. The additional analysis of the validation samples using an alternative MS/MS ion transition for each analyte demonstrated a suitable confirmatory technique (cyclaniliprole *m/z* 602→177, NK-1375 *m/z* 566→266).

An independent laboratory validation [Schoenau, 2013, report 120464] of the method "Determination of cyclaniliprole and NK-1375 in grapes, wine, peaches, oilseed rape seed and dry beans" [Brewin, 2012, report JSM0269] was performed in almonds, apples, lettuce, and wheat. The apples, leaf lettuce and wheat forage were analysed according to the procedures described in the method above (excluding the steps for dry or "oily" matrices). The initial method trials for wheat straw and wheat grain followed the procedure designed to extract the analytes out of matrices with low water content (i.e. dry matrices). The almond nutmeat trial was analysed following the method designed with separate additional steps for "oily" matrices. In order to successfully complete the second method trials minor modifications of the dry matrix method and "oily" matrix method were made for wheat straw and almond nutmeat, respectively. The modification for almond nutmeat and wheat straw consisted of a reduction of the amount of matrix from 20 g to 5 g and an adjustment of the dilution factor of the PE eluate. The method was validated at 0.01 and 0.05 mg/kg for detection of both analytes in the matrix samples tested. Validation results are summarised in Table 45 and Table 46. The validation of the methodology for the determination of cyclaniliprole or NK-1375 in the six matrix types demonstrated that they could be accurately determined at 0.01 and 0.5 mg/kg. The additional analysis of the validation samples using an alternative MS-MS ion transition for each analyte demonstrated a suitable confirmatory technique (cyclaniliprole *m/z* 602→177, NK-1375 *m/z* 566→266). The LOQ was 0.01 mg/kg.

A radiovalidation study [Unsworth, 2014b, report JSM0492] was carried out to validate the efficiency of extraction and analysis of cyclaniliprole and its metabolite NK-1375 using the residue analytical method JSM0269. Samples of lettuce (surface washed lettuce) from the plant metabolism study [Crowe, 2013b, JSM0054] that had been stored frozen (at <-18 °C for at least 5 months) were extracted and analysed using the residue analytical method JSM0269 [Brewin, 2012, report JSM0269]. Solvent extracts from this method were also analysed by the radioanalytical methods used in the lettuce metabolism study [Crowe, 2013b, JSM0054]. Results from the different methods were then compared. Concentrations of radioactivity in solvent extracts produced from residue method JSM0269 were determined by liquid scintillation counting (LSC). Subsamples of the extracts (500 mL) were concentrated to approximately 2 mL. The samples were then analysed by LC and TLC using the methods derived from study JSM0054 [Crowe, 2013b]. The TRR in the surface washed lettuce sample was 0.116 mg/kg. The mean extraction efficiency of total radioactive residues was 62.5% TRR (0.0725 mg/kg). The extraction efficiency was 74.2% TRR determined by the residue method (JSM0269) and radioanalysis in lettuce plants. The relative extraction efficiency of the residue analytical method to the extraction method from the metabolism study was 84.2%. Results are presented in Table 43. A summary of the mean values for cyclaniliprole and its metabolite NK-1375 by the residue analytical method and by radio-HPLC and radio-TLC is shown in Table 44.

Table 43 Comparison of the extraction efficiency of the residue analytical method with the method used in the metabolism study in lettuce

	Extraction efficiency [%]		
	Residue analytical method	Metabolism study method [a]	Relative extraction efficiency
Surface washed lettuce	62.5%	74.2%	84.2%

[a] Neutral-solvents extracts

Table 44 Mean residues of parent cyclaniliprole and metabolite NK-1375 in lettuce extracts in mg/kg analyte

Compound	LC-MS/MS	radio-HPLC	Radio-TLC
Cyclaniliprole	0.05	0.054	0.053
NK-1375	< 0.01	0.014	0.012
TRR in extract	-	0.725	0.0725

LC-MS/MS method JSM0269 is considered valid for determination of cyclaniliprole and metabolite NK-1375 (full validation) in pome fruit, stone fruit, grapes, brassica's, fruiting vegetables, leafy vegetables, soybeans, potatoes, almond, pecan, tea and various processed commodities. The valid LOQ is 0.01 mg/kg (no validations below this level) for both analytes. The crop (groups) covered the crop coups with high acid content, high water content, high oil content, high protein content, high starch content.

LC-MS/MS Method JP2012C106 for plant matrices

For the Japanese studies the same extraction procedure was applied as for method JSM0269. Purification was performed through polymer mini-column followed by anion exchange mini-column. After pre-treatment with acetonitrile and water, water was added to the extract and the mixture was loaded and the polymer column was washed with acetonitrile: water (60:40 v:v) followed by drying. Elution of the column with acetonitrile followed. The eluate was passed through the anion exchange mini-column, which was pre-treated with acetonitrile. Additional acetonitrile was passed and eluates collected. The extracts were subsequently analysed by LC-MS/MS (cyclaniliprole *m/z* 602>284, NK-1375 *m/z* 566>498).

This method was used in the Japanese field residue trials on cherries [Kuzaki and Naruto, 2013, report no code], grapes [Kimikazu, 2013a JP2012C100 and Kimikazu, 2013b, JP2013C280], Chinese cabbage [Yoshiyuki T, 2013c, report JP2012C108, and Hitoshi I, 2013, report JP2013C091], peppers (red) [Cho, 2013, no code;] and (cherry) tomatoes [Yoshiyuki, 2013a, JP2012C105; Yoshiyuki, 2013b, JP2012C106; Kouij, 2012, JP2011C132] and soya bean [Takashi N, 2012a, report JP2011C362 and Yoshiyuki T, 2013d, report JP2012C103, Takashi N, 2012b, report JP2011C361 and Yoshiyuki T, 2013e, report JP2012C102].

Reviewer's conclusion: LC-MS/MS method JP2012C106 is considered valid for determination of cyclaniliprole and metabolite NK-1375 (full validation) in cherries, grapes, Chinese cabbage, peppers, tomatoes, and soybeans. The valid LOQ is 0.01 mg/kg (no validations below this level) for both analytes.

LC-MS/MS Method JP2011C133 for tea and infusions

Samples of crude tea were pulverised and was subsequently swelled for 2 hours with 20 mL water and extracted with 150 mL acetonitrile by shaking. For analysis of hot-water infusion, 360 mL boiling water was added to 6 g intact tea samples and allowed to stand for 5 minutes and filtered. Extracts from pulverised crude tea leaves and hot-water infusion were subjected to the same analytical procedure. Extracts were sequentially purified with polymer adsorbent mini-column (InertSep PLS-2, 1000 mg/6 mL) and anion exchange mini-column (InertSep SAX, 500 mg/6 mL). Cyclaniliprole and NK-1375 were eluted with acetonitrile and diluted further with acetonitrile. The concentrations in the purified fraction were determined with a liquid chromatography and mass spectrometry system (LC-MS/MS) with positive-ion electrospray ionisation (ESI). Quantification of the analytes was achieved by comparison with mixed external standards of cyclaniliprole and NK-1375. The method was validated within the study JP2011C133 [Koki, M. 2012] and also used in Yoshiyuki, 2013f [report JP2012C101].

Reviewer's conclusion: LC-MS/MS method JP2011C133 is considered valid for determination of cyclaniliprole and metabolite NK-1375 (full validation) for tea and infusions. The valid LOQ is 0.01 mg/kg (no validations below this level) for both analytes.

Table 45 Validation results for LC-MS/MS method in plant commodities for determination of parent cyclaniliprole

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
almonds, nutmeat	0.01	0.01	72 69-76	3.8	5	< 0.01	external standard 0.026 -10.4 ng/mL,	120464
		0.5	80 75-84	4.8	5		linear r=0.9997	(ILV of method JSM0269)
almond, nutmeat	0.01	0.01	87 76-100	13.95.1	3	< 0.01	external standards in	IB-2012-JLW-01901-01
		0.2	104 98-108		3		untreated controls	(MV)
almond, nutmeat	0.01	0.01	93 88, 98	na	2	< 0.01	external standards in	IB-2012-JLW-01901-01
		0.2	93 91, 95	na	2		untreated controls	(concurrent recovery)
almond, nutmeat	0.01	0.01	89 76-100	11	5	< 0.01	external standards in	IB-2012-JLW-01901-01
		0.2	100 91-108	7.3	5		untreated controls	(MV&concur. recovery)
almond hulls	0.01	0.01	79 77-86	5.7%5.1	4	< 0.01	external standards in	IB-2012-JLW-01901-01
		0.2	78 74-82	2.5	3		untreated	(method validation)
		3	83 81-85		3		controls	
almond, hulls	0.01	0.01	82 76-91	8	6	< 0.01	external standards in	IB-2012-JLW-01901-01
		0.2	82 75-88	5.4	6		untreated controls	(concurrent recovery)
almond, hulls	0.01	0.01	81 76-91	7	10	< 0.01	external standards in	IB-2012-JLW-01901-01
		0.2	80 74-88	5.5	9		untreated controls	(MV&concur. recovery)
apples	0.01	0.01	95 89-101	4.9	5	< 0.01	external standard 0.026 -10.4 ng/mL,	120464
		0.5	95 93-96	1.5	5		linear, r=1.000	(ILV of method JSM0269)
apple fruit	0.01	0.01	86 71-95	119	6	< 0.01	external std	JSM0347
		0.5	80 71-90		6		in solvent 0.025-10 ng/mL linear	(procedural recovery)
apple fruit	0.01	0.01	103 98-107	3.7%3.2	4	< 0.01	external std	JSM0348
		0.5	91 87-93		3		in solvent 0.025-10 ng/mL linear	(procedural recovery)
apple fruit	0.01	0.01	98 95-99	2.30%	3	< 0.01	external std	JSM0473
		0.5	93 90-99	4.92%	3		in solvent 0.025-10 ng/mL linear	(procedural recovery)
apple fruit	0.01	0.01	93 73-112	11.30%	8	< 0.01	external std	IB-2012-JLW-020
		0.5	104 97-109	4.70%	6		in solvent	(MV& concur. recovery)
		1	102 96-108	na	2		0.025-10	

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
							ng/mL linear	
apple juice	0.01	0.01 4.1	108 101-112 109 102-121	4.60% 8.00%	4 4	< 0.01	idem	idem
apple wet pomace	0.01	0.01 4.1	99 na 98 na	na	1 1	< 0.01	idem	idem
apple fruit	0.01	0.01 0.1 0.2	90 87, 93 100 98, 102 101 100-102	na na 1.10%	2 2 3	< 0.01	external standards in untreated controls	IB-2012-JLW-004 (concurrent recovery)
apple fruit	0.01	0.01 0.1	103 nr 98 nr	6.30% 8.10%	5 5	< 0.01	external standards in untreated controls 0.05-50 µg/L linear	UPL-113 (concurrent recovery)
apple fruit	0.01	0.01 0.1	105 nr 103 nr	2.40% na	5 2	< 0.01	external standards in untreated controls 0.05-50 µg/L linear	ISK12433 (concurrent recovery)
apple[a]	0.01	0.01 1	112 99, 104 nr 85 84, 86	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL Linear, r≥0.997	SQ74KP (procedural recovery)
apple, peel	0.01	1 5	85 nr 101 nr	na	1 1	< 0.01	idem	idem
apple, canned fruit	0.01	0.1	na 92	na	1	< 0.01	idem	idem
apple, fruit syrup	0.01	0.01	na 100	na	1	< 0.01	idem	idem
apple, solid parts (after sieving)	0.01	0.01 5	na 103 na 77	na na	1 1	< 0.01	idem	idem
apple sauce, before and after pasteurization	0.01	0.01 1	95 90, 100 92 92, 93	na na	1 1	< 0.01	idem	idem
apple, (raw) juice	0.01	0.1	97 96, 102	na	2	< 0.01	idem	idem
apple, wet pomace	0.01	0.1 5	na 91 na 93	na na	1 1	< 0.01	idem	idem
apple, dry pomace	0.01	1 5	na 106 na 93	na na	1 1	< 0.01	idem	idem
apple, jelly	1	1	na 100	na	1	< 0.01	idem	idem
apricot flesh	0.01	0.01 0.5	96 90-107 86 83-88	9% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0329 (procedural recovery)
apricot flesh	0.01	0.01 0.5	90 88-92 96 95-96	2% 1%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0474 (procedural recovery)
apricot flesh	0.01	0.01 0.5	86 77-95 90 82-97	9% 8%	3 3	< 0.01	external std in solvent	JSM0667 (procedural recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
							0.025-10 ng/mL linear	
beans, dry	0.01	0.01 0.5	92 84-97 91 88-95	5.80% 3.80%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r= 0.9985	JSM0269 (method validation)
broccoli	0.01	0.01 0.5	91 86-99 77 75-79	7% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0333 (procedural recovery)
broccoli	0.01	0.01 0.5	89 72-92 90 87-92	13% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0481 (procedural recovery)
broccoli	0.01	0.01 0.1 1	94 88-100 na 107 96 87-106	5.80% - 8.30%	5 1 4	< 0.01	external standards in untreated controls	IB-2012-JLW-28-01-01 (concurrent recovery)
broccoli	0.01	0.01 0.1	94 91-96 97 95-99	2.50% 2.40%	3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-28-01-01 (method validation)
Brussels sprouts	0.01	0.01 0.5	93 92-94 83 82-86	1% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0340 (procedural recovery)
Brussels sprouts	0.01	0.01 0.5	91 85-101 90 89-92	9% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0484 (procedural recovery)
Brussels sprouts	0.01	0.01 0.5	86 76-100 85 82-87	12% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0603 (procedural recovery)
cabbage, head	0.01	0.01 0.5	99 94-102 80 77-83	4% 2%	5 5	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0334 (procedural recovery)
cabbage, head	0.01	0.01 0.5	91 74-102 92 88-95	12% 3%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0482 (procedural recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
cabbage, head	0.01	0.01 0.1 0.5	104 97-108 na 92 96 90-100	5.00% - 5.60%	4 1 3	< 0.01	external standards in untreated controls	IB-2012-JLW-028-01-01 (concurrent recovery)
cantaloupe	0.01	0.01 0.1	98 88-109 100 93-107	9.20% 5.70%	4 4	< 0.01	external standards in untreated controls	IB-2013-JAM-003 (concurrent recovery)
cauliflower	0.01	0.01 0.5	85 77-93 75 73-80	7% 3%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0332 (procedural recovery)
cauliflower	0.01	0.01 0.5	104 99-109 86 85-88	5% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0480 (procedural recovery)
cherries, sweet	0.01	0.01 0.1 0.5	86 74-104 90 84-101 99 97-103	4.30% 13% 10.90%	6 3 3	< 0.01	external standards in untreated controls	IB-2013-JLW-005 (concurrent recovery)
cherries, tart	0.01	0.01 0.1 0.8	99 94-105 100 99-101 93 na	5.50% 0.80% na	4 3 1	< 0.01	external standards in untreated controls	IB-2013-JLW-005 (concurrent recovery)
cherries	0.01	0.01 0.5	81 77-84 94 93-96	3.40% 1.10%	6 6	< 0.01	external standards 0.05-2 µg/L	No code (Kuzaki &Naruto, 2013 (concurrent recovery)
Chinese cabbage	0.01	0.01 0.5	98 87-104 96 93-100	5.80% 2.60%	10 10	< 0.01	external standards in untreated controls	JP2013C091 (concurrent recovery)
Chinese cabbage	0.01	0.01 0.5	98 87-110 92 87-99	7.40% 4.70%	10 10	< 0.01	external standards in untreated controls	JP2012C108 (concurrent recovery)
cucumber	0.01	0.01 0.1	96 90-107 102 98-109	7.70% 4.80%	4 4	< 0.01	external standards in untreated controls	IB-2013-JAM-003 (concurrent recovery)
grapes	0.01	0.01 0.5	99 93-101 92 90-94	3.30% 1.60%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9985	JSM0269 (ILV)
grapes	0.01	0.01 0.1 0.5 0.8	97 89-100 -92 92 87-97 99 94-104	7.60% - 4.00% 4.90%	6 1 5 3	< 0.01	external std in solvent, 0.025-2.5 ng/mL, linear	IB-2013-JAM-002 (concurrent recovery)
grapes	0.01	0.05 0.1	100 85-116 101 99-103	12.40% 1.60%	5 5	< 0.01	external standard in untreated controls, linear, r=	JP2012C100A (method recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
							0.9999	
grapes	0.01	0.05 0.1	103 91-119 96 94-98	10.40% 1.70%	5 5	< 0.01	external standard in untreated controls, linear, r= 0.9999	JP2012C100B (method recovery)
grapes	0.01	0.05 0.1	97 89-104 99 97-100	6.40% 1.30%	5 5	< 0.01	external standard in untreated controls, linear, r= 0.9999	JP2012C100C (method recovery)
grapes	0.01	0.05 0.1	90 88-92 74 71-79	2.00% 4.90%	5 5	< 0.01	external standard in untreated controls, linear, r= 0.9999	JP2013C280 (method recovery)
grapes, wine	0.01	0.01 0.5	94 84-109 91 88-93	10.40% 2.30%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9985	JSM0269 (method validation)
grapes, processed to wine	0.01	0.01 0.5	101 90-108 85 72-91	8% 9%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0349 (procedural recovery)
grapes bunches	0.01	0.01 0.5	105 104,106 92 91, 92	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9992	JSM0350 (procedural recovery)
grapes, wet pomace	0.01	0.01 0.5	100 97-106 84 79-88	4.00% 4.50%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9992	JSM0350 (procedural recovery)
grapes, wine	0.01	0.01 0.5	90 82-98 88 82-93	8.90% 6.30%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9992	JSM0350 (procedural recovery)
grapes, raisins	0.01	0.01 0.5	na 103 na 82	na na	1 1	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9992	JSM0350 (procedural recovery)
grapes, clear juice	0.01	0.01	na 106	na	1	< 0.01	external std in solvent	JSM0350 (procedural

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
							0.025-10 ng/mL linear, r=0.9992	recovery)
grapes, wine (RAC)	0.01	0.01 0.5	98 94-100 91 89-92	3% 2%	4 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0478 (procedural recovery)
grapes, table	0.01	0.01 0.5	95 87-105 78 74-82	9% 4%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0330 (procedural recovery)
grapes, table	0.01	0.01 0.5	99 95-102 93 91-95	4% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0477 (procedural recovery)
kale	0.01	0.01 0.5	94 89-99 85 82-91	5% 4%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0483 (procedural recovery)
lettuce, head	0.01	0.01 0.1 0.2 1 2.4	103 878-114 107 105,108 102 100,104 na 96 103 97-108	8.20% na na na 5%	8 2 2 1 3	< 0.01	external standard in untreated controls	IB-2012-JAM-0010-01-01 (concurrent recovery)
lettuce, leaf	0.01	0.01 0.1 2.4 4.5	98 94-108 na 99 na 105 101 95-109	5.30% na na 6.00%	6 1 1 4	< 0.01	external standard in untreated controls	IB-2012-JAM-0010-01-01 (concurrent recovery)
spinach	0.01	0.01 0.1 3.7 4.1 5	94 88-98 na 99 na 101 na 94 90 87,94	5.10% na na na na	5 1 1 1 2	< 0.01	external standard in untreated controls	IB-2012-JAM-0010-01-01 (concurrent recovery)
lettuce, leaf	0.01	0.01 0.5	89 86-91 91 90-92	2.60% 1.00%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, linear, r= 0.9999	120464 (ILV of method JSM0269)
mustard greens	0.01	0.01 0.1 5.7	100 94-106 102 102-103 106 104-107	0.70% na na	4 2 2	< 0.01	external standard in untreated controls	IB-2012-JLW-028-01-01 (concurrent recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
mustard greens	0.01	8.1	96 94-97	1.50%	3	< 0.01	external standard in untreated controls	IB-2012-JLW-028-01-01 (method validation)
oilseed rape seed	0.01	0.01 0.5	101 99-106 89 84-93	2.70% 3.70%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r= 0.9985	JSM0269 (method validation)
peaches, flesh	0.01	0.01 0.5	99 97-100 76 74-77	2% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0351 (procedural recovery)
peaches, flesh	0.01	0.01 0.5	99 97,101 84 81-86	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0352 (procedural recovery)
peaches, canned/puree	0.01	0.01 0.5	100 98, 101 80 80, 81	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0352 (procedural recovery)
peaches, flesh	0.01	0.01 0.5	89 86-95 94 91-99	5% 4%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0475 (procedural recovery)
peaches, flesh	0.01	0.01 0.1 0.4	99 85-103 99 93-104 92 90-93	3.50% 5.90% 1.80%	6 3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-005 (concurrent recovery)
peaches	0.01	0.01 0.5	104 93-109 96 94-98	6.70% 1.60%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r= 0.9985	JSM0269 (method validation)
peaches, fruit w/o stones	0.01	1	na 94	na	1	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9998	QK27SS (method recovery)
peaches, blanched/pureed/canned	0.01	0.01 0.1 1	104 100,109 na 93 85 80-93	na na 6.10%	2 1 5	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9998	QK27SS (method recovery)
peaches, peeled fruit	0.01	0.01 0.1	na 110 na 88	na na	1 1	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9998	QK27SS (method recovery)
peaches, (raw) juice	0.01	0.01 1	93 87, 99 96 94, 97	na na	2 2	< 0.01	external standard	QK27SS (method recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
							0.025 to 5 ng/mL, linear, r=0.9998	
peaches, jam	0.01	0.01 1	na 95 na 86	na	1	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9998	QK27SS (method recovery)
pear fruit	0.01	0.01 0.1 0.2	94 87-109 100 99-101 99 97-101	9.97% 1.10% 2.90%	5 3 2	< 0.01	external standards in untreated controls	IB-2012-JLW-004 (concurrent recovery)
pecan, nutmeat	0.01	0.01 0.2	91 84-100 102 101-102	8.80% 0.60%	3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-019-01-01 (MV)
pecan, nutmeat	0.01	0.01 0.2	83 81, 85 87 84, 90	na na	2 2	< 0.01	external standards in untreated controls	IB-2012-JLW-019-01-01 (concurrent recovery)
pecan, nutmeat	0.01	0.01 0.2	88 81-100 96 84-102	8.50% 8.70%	5 5	< 0.01	external standards in untreated controls	IB-2012-JLW-019-01-01 (MV+concurrent recovery)
plum flesh	0.01	0.01 0.5	98 90-103 86 75-88	5% 5%	6 6	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0338 (procedural recovery)
plum flesh	0.01	0.01 0.5	97 94-99 94 92-95	3% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0476 (procedural recovery)
plum flesh	0.01	0.01 0.1 1	94 90-96 102 91 88-95	2.90% na 3.70%	4 1 3	< 0.01	external standards in untreated controls	IB-2013-JLW-005-01-01 (concurrent recovery)
prunes	0.01	0.01 0.1 4.7	86 74-96 89 82-98 100 99-102	10.60% 8.50% 1.59%	4 4 3	< 0.01	external standards in untreated controls	IB-2013-JLW-005-01-01 (concurrent recovery)
red pepper, fruit	0.1	0.1 0.5	76 75-78 87 86-88	2.30% 1.40%	3 3	< 0.01	external standard in controls	Cho, 2013, no code (concurrent recovery)
red pepper, leaves	0.1	0.1 0.5	74 74-76 72 72 (3x)	1.80% 0.30%	3 3	< 0.01	external standard in controls	Cho, 2013, no code (concurrent recovery)
soy bean, dried	0.01	0.01 0.5	98 95-104 98 96-99	3.50% 1.50%	6 6	< 0.01	external standard in untreated controls	JP2011C361 (concurrent recovery)
soy bean, green	0.01	0.01 0.5	91 90-93 98 91-100	1.50% 3.50%	6 6	< 0.01	external standard in untreated controls	JP2011C362 (concurrent recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
summer squash	0.01	0.01 0.1	102 94-109 102 97-106	6.10% 3.50%	8 8	< 0.01	external standards in untreated controls	IB-2013-JAM-003 (concurrent recovery& method validation)
sweet pepper	0.01	0.01 0.5	104 96-110 93 69-92	7% 9%	5 5	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0337 (procedural recovery)
sweet pepper	0.01	0.01 0.1 0.5	90 86-95 91 87-93 95 92-97	5% 3% 3%	3 3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0487 (procedural recovery)
sweet pepper	0.01	0.01 0.5	94 82-107 80 73-90	8% 6%	8 7	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0336 (procedural recovery)
sweet pepper	0.01	0.01 0.1 0.5	98 93-10 93 87-98	5% 3% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0485 (procedural recovery)
sweet pepper (bell)	0.01	0.01 0.1 0.2	106 101-116 na 108 98 92-104	6.60% na 5.90%	4 1 3	< 0.01	external standards in untreated controls	IB-2012-JLW-029-01-01 (concurrent recovery)
sweet pepper (non-bell)	0.01	0.01 0.1	102 88-112 102 98-109	10.10% 3.90%	5 5	< 0.01	external standards in untreated controls	IB-2012-JLW-029-01-01 (concurrent recovery)
tea, crude tea	0.02	0.02 2 10	93 90-95 98 93-105 91 86-95	2.20% 6.80% 3.90%	6 6 6	< 0.02	external std in solvent, linear, r=0.999	JP2011C133 (method recovery)
tea, infusion	0.02	0.02 2	86 84-88 89 88-89	2.10% 0.60%	6	< 0.02	external std in solvent, linear, r=0.999	JP2011C133 (method recovery)
tea, crude	0.02	0.2	89 85-92	4.10%	3	< 0.02	spiked control samples	JP2011C133 (quality control)
tea, infusion	0.02	0.2	90 90, 91	n.a.	2	< 0.02	spiked control samples	JP2011C133 (quality control)
tomato	0.01	0.01 0.5 1	97 71-108 84 81-94 n.a. 83	13% 5% n.a.	7 6 1	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0335 (procedural recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
tomato	0.01	0.01 0.5	94 90-101 91 89-95	5% 3%	5 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0486 (procedural recovery)
tomato	0.01	0.01 0.5	103 100-105 87 86-87	3% 1%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0353 (procedural recovery)
tomato	0.01	0.01 0.5	99 106, 92 86 85, 88	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0354 (procedural recovery)
tomato	0.01	0.01 1	na 72 na 99	na na	1 1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, blanched	0.01	0.01 1	na 73 na 98	na na	1 1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, canned	0.01	1	na 91	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, peeled	0.01	0.01	na 70	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, dried	0.01	5	na 79	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, raw juice and juice	0.01	1	98 100, 95	na	2	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, ketchup, paste, puree	0.01	0.01 1 5	na 100 91 80-110 na 96	na 15.70% na	1 3 1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
tomato, vegetable stock	0.01	0.01	na 108	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, dry and wet pomace	0.01	5 15	96 91, 100 90 87, 94	na na	2 2	< 0.01	external standard in solvent 0.025-10 ng/mL linear r=09988	HH97BD (procedural recovery)
tomato, canned	0.01	0.01 0.5	108 108, 108 90 94, 87	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0354 (procedural recovery)
tomato, (raw) juice	0.01	0.01 0.5	94 97, 90 88 85, 91	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0354 (procedural recovery)
tomato	0.01	0.01 0.5	101 97-105 97 91-100	4% 5%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0488 (procedural recovery)
tomato	0.01	0.01 0.5	88 86-94 107 103-109	3.70% 2.20%	5 5	< 0.01	spiked samples linear, r=0.999	JP2012C105 (method recovery)
tomato	0.01	0.01 0.5	85 78-98 97 96-99	8.90% 1.10%	5 5	< 0.01	spiked samples, linear, r=0.999	JP2012C106 (method recovery)
tomato	0.01	0.01 0.5	73 70-78 101 97-105	4.40% 3.20%	6 6	< 0.01	spiked samples, linear, r=0.999	JP2011C132 (method recovery)
tomato	0.01	0.01 0.1	99 90-110 99 91-105 93 92-96	6.20% 5.10% 2.50%	9 6 3	< 0.01	external standard, linear, r ≥ 0.993	IB-2012-JLW-029-01-01 (concurrent recovery)
tomato, puree	0.01	0.01 0.1	99 96-105 98 95-101	4.10% 2.80%	4 4	< 0.01	external standard, r= 0.9997	IB-2012-JLW-029-01-01 (method validation, including 1 concurrent)
tomato paste	0.01	0.01 0.1	96 93-103 95 91-95	4.3 1.70%	4 4	< 0.01	external standard, r= 1.000	IB-2012-JLW-029-01-01 (method validation, including 1 concurrent recovery)

Matrix	Reported LOQ	Fortification level (mg/kg)	Recovery (%) mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
wheat, forage	0.01	0.01	88 87-91	1.70%	5	< 0.01	external standard 0.026 -10.4 ng/mL,	120464
		0.5	92 91-94	1.40%	5		linear, r= 0.9999	(ILV of method JSM0269)
wheat, forage	0.01	0.01	97	n.a.	1	< 0.01	External std in solvent	JSM0414
		0.5	77	n.a.	1		0.025 – 10 ng/mL linear r=0.9989 (P)	(procedural recovery)
wheat, forage	0.01	0.01	90 78-97	7.60%	6	< 0.01	External std in solvent	IB-2012-JLW-022-01-01
		0.1	93 88-99	4.50%	6		0.025 – 2.5 ng/mL linear, r≥0.9994 (P)	(procedural recovery)
wheat, grain	0.01	0.01	87 80-91	4.80%	5	< 0.01	external standard 0.026 -10.4 ng/mL,	120464
		0.5	92 91-94	1.00%	5		linear, r= 0.9999	(ILV of method JSM0269)
wheat, grain	0.01	0.01	94	n.a.	1	< 0.01	External std in solvent	JSM0414
		0.5	102	n.a.	1		0.025 – 10 ng/mL linear r=0.9989 (P)	(procedural recovery)
wheat, grain	0.01	0.01	88 78-102	9.70%	6	< 0.01	External std in solvent	IB-2012-JLW-022-01-01
		0.1	91 84-97	4.80%	6		0.025 – 2.5 ng/mL linear r≥0.9994 (P)	(procedural recovery)
wheat, hay	0.01	0.01	90	n.a.	1	< 0.01	External std in solvent	JSM0414
		0.25	87	n.a.	1		0.025 – 10 ng/mL linear	(procedural recovery)
		0.5	88	n.a.	1		r=0.9989 (P)	
wheat, straw	0.01	0.01	84 82-86	2.20%	5	< 0.01	external standard 0.026 -10.4 ng/mL,	120464
		0.5	86 85-88	1.10%	5		linear, r= 0.9999	(ILV of method JSM0269)
wheat, straw	0.01	0.01	98 89-106	7.50%	6	< 0.01	External std in solvent	IB-2012-JLW-022-01-01
		0.1	93 90-95	2.80%	5		0.025 – 2.5 ng/mL	(procedural recovery)
		0.2	96	n.a.	1		linear, r≥0.9994 (P)	

[a] Combined data from apple without stems and apple peeled.

Table 46 Validation results for LC-MS/MS method in plant commodities for metabolite NK-1375

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
original method									
almonds, nutmeat	0.01	0.01 0.5	82 98	78-95 94-101	3.3% 2.6%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, r=0.9999	120464 (ILV of method JSM0269)
almond, nutmeat	0.01	0.01 0.2	91 97	74-105 74-103	17.3% 5.1%	3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-01901-01 (method validation)
almond, nutmeat	0.01	0.01 0.2	109 102	104-114 100,105	4.8% na	4 2	< 0.01	external standards in untreated controls	IB-2012-JLW-01901-01 (concurrent recovery)
almond, nutmeat	0.01	0.01 0.2	101 99	74-114 94-105	13.5% 4.9%	7 5	< 0.01	external standards in untreated controls	IB-2012-JLW-01901-01 (method validation & concurrent recovery)
almond hulls	0.01	0.01 0.2 3.0	86 86 79	76-95 84-89 78-80	9.1% 3.4% 1.3%	4 3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-01901-01 (method validation)
almond, hulls	0.01	0.01 0.2	84 82	70-92 75-90	9.5% 7.7%	6 6	< 0.01	external standards in untreated controls	IB-2012-JLW-01901-01 (concurrent recovery)
almond, hulls	0.01	0.01 0.2	84 83	70-95 75-90	8.9% 6.6%	10 9	< 0.01	external standards in untreated controls	IB-2012-JLW-01901-01 (method validation & concurrent recovery)
apples	0.01	0.01 0.5	92 102	98-94 101-103	2.2% 0.7%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, r= 0.9995	120464 (ILV of method JSM0269)
apple fruit	0.01	0.01 0.5	90 85	75-99 74-88	8% 7%	6 6	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0347 (procedural recovery)
apple fruit	0.01	0.01 0.5	98 85	93-103 84-91	4.6% 3.5%	4 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0348 (procedural recovery)
apple fruit	0.01	0.01 0.5	97 89	89-106 86-94	8.5% 4.2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0473 (procedural recovery)
apple fruit	0.01	0.01 0.5 1.0	88 102 100	76-100 98-106 94-107	9.8% 3.3% na	8 6 2	< 0.01	external std in solvent 0.026-2.6 ng/mL linear	IB-2012-JLW-020 (method validation& concurrent recovery)
apple juice	0.01	0.01 4.1	107 110	98-112 106-116	6.1% 3.8%	4 4	< 0.01	external std in solvent 0.026-2.6 ng/mL linear	IB-2012-JLW-020 (method validation& concurrent recovery)
apple wet pomace	0.01	0.01 4.1	111 109	na na	na na	1 1	< 0.01	external std in solvent	IB-2012-JLW-020

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean range		RSD (%)	n	control mg/kg	calibration	Code no; Report no
								0.026-2.6 ng/mL linear	(method validation& concurrent recovery)
apple fruit	0.01	0.01 0.1 0.2	106 98 99	111, 100 95, 101 99-100	na na 0.3%	2 2 3	< 0.01	external standards in untreated controls	IB-2012-JLW-004 (concurrent recovery)
apple fruit	0.01	0.01 0.1	89 92	nr nr	13% 7.3%	5 5	< 0.01	external standards in untreated controls 0.05-50 µg/L linear	UPL-113 (concurrent recovery)
apple fruit	0.01	0.01 0.1	85 97	nr nr	5.4% na	5 2	< 0.01	external standards in untreated controls 0.05-50 µg/L linear	ISK12433 (concurrent recovery)
apple[a]	0.01	0.01	100	88,112	na	2	< 0.01	external std in solvent 0.025-10 ng/mL Linear, r≥0.997	SQ74KP (procedural recovery)
apple, peel	0.01	1 5	na na	88 99	na na	1 1	< 0.01	Idem	Idem
apple, core and stalks	0.01	0.01	na	92	na	1	< 0.01	Idem	Idem
apple, peeled, without core and stalks	0.01	0.1	na	91	na	1	< 0.01	Idem	Idem
apple, canned fruit	0.01	0.1	na	93	na	1	< 0.01	Idem	Idem
apple, stalks	0.01	1 5	na na	75 98	na na	1 1	< 0.01	Idem	Idem
apple, fruit syrup	0.01	0.01	na	101	na	1	< 0.01	Idem	Idem
apple, solid parts (after sieving)	0.01	0.01	na	107	na	1	< 0.01	Idem	Idem
apple sauce, before pasteurization	0.01	0.01	na	105	na	1	< 0.01	Idem	Idem
apple sauce, after pasteurization	0.01	0.1	na	93	na	1	< 0.01	Idem	Idem
apple, raw juice	0.01	0.1	96	94, 97	na	2	< 0.01	Idem	Idem
apple, juice	0.01	1	na	97	na	1	< 0.01	Idem	Idem
apple, wet pomace	0.01	0.1 5	na na	95 97	na na	1 1	< 0.01	Idem	Idem
apple, dry pomace	0.01	1 5	na na	92 90	na na	1 1	< 0.01	Idem	Idem
apple, jelly	0.01	1	na	96	na	1	< 0.01	Idem	Idem
apricot flesh	0.01	0.01 0.5	99 88	97-101 84-92	2% 4%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0329 (procedural recovery)
apricot flesh	0.01	0.01 0.5	101 100	95-104 99-102	5% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0474 (procedural recovery)
apricot flesh	0.01	0.01 0.5	103 97	96-109 88-101	7% 8%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0667 (procedural recovery)
beans, dry	0.01	0.5	98 91	86-109 92-97	9.6% 2.3%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9991	JSM0269 (method validation)
broccoli	0.01	0.01 0.5	99 79	89-109 76-81	10% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0333 (procedural recovery)

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
broccoli	0.01	0.01 0.5	95 89	89-104 84-98	8% 8%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0481 (procedural recovery)
broccoli	0.01	0.01 0.1 1.0	93 na 97	87-101 100 91-106	6.1% - 6.9%	5 1 4	< 0.01	external standards in untreated controls	IB-2012-JLW-028-01-01 (concurrent recovery)
broccoli	0.01	0.01 0.1	102 98	97-106 97-101	4.4% 2.3%	3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-028-01-01 (method validation)
Brussels sprouts	0.01	0.01 0.5	97 83	92-104 82-86	6% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0340 (procedural recovery)
Brussels sprouts	0.01	0.01 0.5	101 86	100-104 83-88	2% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0484 (procedural recovery)
Brussels sprouts	0.01	0.01 0.5	87 80	84-92 79-80	4% 0.6%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0603 (procedural recovery)
cabbage, head	0.01	0.01 0.5	101 78	95-105 76-81	4% 2%	5 5	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0334 (procedural recovery)
cabbage, head	0.01	0.01 0.5	96 89	91-98 82-101	4% 8%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0482 (procedural recovery)
cabbage, head	0.01	0.01 0.1 0.5	105 na 96	96-109 93 93-99	5.6% - 3.6%	4 1 3	< 0.01	external standards in untreated controls	IB-2012-JLW-028-01-01 (concurrent recovery)
cantaloupe	0.01	0.1	97 99	82-107 95-105	10.1% 4.6%	4 4	< 0.01	external standards in untreated controls	IB-2013-JAM-003-01-01 (concurrent recovery)
cauliflower	0.01	0.01 0.5	94 77	78-108 74-80	10% 3%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0332 (procedural recovery)
cauliflower	0.01	0.01 0.5	88 85	78-97 80-92	10% 6%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0480 (procedural recovery)
cherries, sweet	0.01	0.01 0.1 0.5	84 88 97	76-101 81-100 95-101	11.8% 12% 3.4%	6 3 3	< 0.01	external standards in untreated controls	IB-2013-JLW-005 (concurrent recovery)
cherries, tart	0.01	0.01 0.1 0.8	97 97 95	89-105 94-100 na	8.1% 3.1% na	4 3 1	< 0.01	external standards in untreated controls	IB-2013-JLW-005 (concurrent recovery)
cherries	0.01	0.01 0.5	85 86	84-88 84-89	2.1% 2.3%	6 6	< 0.01	external standards 0.05-2 µg/L	JP2012C100 (concurrent recovery)
Chinese cabbage	0.01	0.01 0.5	97 97	88-108 93-104	7.8% 4.6%	10 10	< 0.01	external standards in untreated controls	JP2013C091 (concurrent recovery)

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
Chinese cabbage	0.01	0.01 0.5	96 93	87-109 89-98	6.9% 3.3%	10 10	< 0.01	external standards in untreated controls	JP2012C108 (concurrent recovery)
cucumber	0.01	0.1	101 100	90-117 94-106	11.1% 5.4%	4 4	< 0.01	external standards in untreated controls	IB-2013-JAM-003-01-01 (concurrent recovery)
grapes	0.01	0.5	97 94	86-109 92-97	8.8% 2.3%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9991	JSM0269 (ILV)
grapes	0.01	0.01 0.1 0.5 0.8	96 na 92 96	87-104 95 85-100 92-101	9.7% na 6.2% 5.1%	6 1 5 3	< 0.01	external std in solvent, 0.025-2.5 ng/mL, linear	IB-2013-JAM-002 (concurrent recovery)
grapes	0.01	0.05 0.1	114 102	108-117 100-104	4.3% 1.4%	5 5	< 0.01	external standard in untreated controls, linear, r ≥ 0.9996	JP2012C100A (method recovery)
grapes	0.01	0.05 0.1	94 97	84-99 96-99	6.3% 1.3%	5 5	< 0.01	external standard in untreated controls, linear, r ≥ 0.9996	JP2012C100B (method recovery)
grapes	0.01	0.05 0.1	102 102	90-112 100-104	9.5% 1.8%	5 5	< 0.01	external standard in untreated controls, linear, r ≥ 0.9996	JP2012C100C (method recovery)
grapes	0.01	0.05 0.1	89 79	88-90 77-82	1.0% 2.4%	5 5	< 0.01	external standard in untreated controls, linear, r ≥ 0.999	JP2013C280 (method recovery)
grapes, processed to wine	0.01	0.01 0.5	94 93	82-109 88-98	12.7% 4.5%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9991	JSM0269 (method validation)
grapes, processed to wine	0.01	0.01 0.5	102 86	99-103 77-100	2% 10%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0349 (procedural recovery)
grapes bunches	0.01	0.01 0.5	92 90	83, 100 86, 95	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9998	JSM0350 (procedural recovery)
grapes, wet pomace	0.01	0.01 0.5	90 84	80-94 78-88	7.2% 5.3%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9998	JSM0350 (procedural recovery)
grapes, wine	0.01	0.01 0.5	93 88	89-95 81-100	3.5% 11.6%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9998	JSM0350 (procedural recovery)
grapes, raisins	0.01	0.01 0.5	na na	108 85	na na	1 1	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9998	JSM0350 (procedural recovery)
grapes, clear juice	0.01	0.01	na	103	na	1	< 0.01	external std in solvent 0.025-10 ng/mL linear, r=0.9998	JSM0350 (procedural recovery)
grapes, wine (RAC)	0.01	0.01 0.5	95 91	89-100 90-93	5% 2%	4 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0478 (procedural recovery)
grape, table	0.01	0.01 0.5	100 78	96-105 73-80	5% 4%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL	JSM0330 (procedural recovery)

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
								linear	
grapes, table	0.01	0.01 0.5	102 93	96-109 91-94	7% 2%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0477 (procedural recovery)
kale	0.01	0.01 0.5	101 81	97-108 78-89	6% 5%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0483 (procedural recovery)
lettuce, head	0.01	0.01 0.1 0.2 0.4 1.0	101 103 104 na na	93-112 100,105 101-105 98 96	7% na 1.9% na na	8 2 4 1 1	< 0.01	external standard in untreated controls	IB-2012-JAM-0010-01-01 (concurrent recovery)
lettuce, leaf	0.01	0.01 0.1 0.25 0.4 0.5	97 na na 100 na	91-104 101 105 91-105 93	4.4% na na 7.6% na	6 1 1 3 1	< 0.01	external standard in untreated controls	IB-2012-JAM-0010-01-01 (concurrent recovery)
spinach	0.01	0.01 0.1 0.4 1.0	99 na na 89	94-103 96 101 92-93	4.2% na na 6.7%	5 1 1 3	< 0.01	external standard in untreated controls	IB-2012-JAM-0010-01-01 (concurrent recovery)
lettuce, leaf	0.01	0.5	89 93	87-93 92-95	2.5% 1.3%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, linear, r= 0.9998	120464 (ILV of method JSM0269)
mustard greens	0.01	0.01 0.1 0.5 0.7	97 104 na na	90-105 103,105 99 106	7.2% na na na	4 2 1 1	< 0.01	external standard in untreated controls	IB-2012-JLW-028-01-01 (concurrent recovery)
oilseed rape seed	0.01	0.02 0.5	98 90	89-105 82-93	7.8% 4.9%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9991	JSM0269 (method validation)
peaches, flesh	0.01	0.01 0.5	100 78	94-104 75-80	5% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0351 (procedural recovery)
peaches, flesh	0.01	0.01 0.5	82 82	77, 87 79, 84	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0352 (procedural recovery)
peaches, canned/puree	0.01	0.01 0.5	96 82	92, 99 82, 83	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0352 (procedural recovery)
peaches, flesh	0.01	0.01 0.5	96 89	90-99 86-91	5% 3%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0475 (procedural recovery)
peaches, flesh	0.01	0.01 0.1 0.4	96 101 92	81-109 97-106 90-95	12.4% 4.7% 3.5%	6 3 3	< 0.01	idem	idem
peaches	0.01	0.5	103 90	99-110 87-95	4.4% 3.8%	5 5	< 0.01	external std in solvent, 0.025-10 ng/mL linear, r=0.9991	JSM0269 (method validation)
peaches, fruit w/o stones	0.01	1	na	94	na	1	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9970	QK27SS (method recovery)
peaches,	0.01	0.01	96	90, 101	na	2	< 0.01	external standard	QK27SS

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
blanched/pureed/canned		0.1 1	na 88	98 86, 89	na na	1 2		0.025 to 5 ng/mL, linear, r=0.9970	(method recovery)
peaches, peeled fruit	0.01	0.01 0.1	na na	114 94	na na	1 1	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9970	QK27SS (method recovery)
peaches, (raw) juice	0.01	0.01	96	93, 99	na	2	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9970	QK27SS (method recovery)
peaches, jam	0.01	0.01 1	na	102	na	1	< 0.01	external standard 0.025 to 5 ng/mL, linear, r=0.9970	QK27SS (method recovery)
pear fruit	0.01	0.01 0.1 0.2	103 104 101	95-113 103-104 98-103	6.4% 0.6% na	5 3 2	< 0.01	external standards in untreated controls	IB-2012-JLW-004 (concurrent recovery)
pecan, nutmeat	0.01	0.01 0.2	96 95	94-100 89-102	3.6% 7.0%	3 3	< 0.01	external standards in untreated controls	IB-2012-JLW-019-01-01 (MV)
pecan, nutmeat	0.01	0.01 0.2	90 100	78-114 95-104	18% 4.4%	4 4	< 0.01	external standards in untreated controls	IB-2012-JLW-019-01-01 (concurrent recovery)
pecan, nutmeat	0.01	0.01 0.2	93 98	78-114 89-104	13% 5.8%	7 7	< 0.01	external standards in untreated controls	IB-2012-JLW-019-01-01 (MV+concurrent recovery)
plum flesh	0.01	0.01 0.5	96 88	82-101 76-92	8% 7%	6 6	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0338 (procedural recovery)
plum flesh	0.01	0.01 0.5	102 94	101-103 94-95	1% 1%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0476 (procedural recovery)
plum flesh	0.01	0.01 0.1 1.0	92 na 92	86-97 98 91-92	4.7% na 0.4%	4 1 3	< 0.01	idem	idem
prunes	0.01	0.01 0.1 0.6	92 90 98	77-108 108 79-100 94-102	14.1% 11.1% 4.3%	4 4 3	< 0.01	external standards in untreated controls	IB-2013-JLW-005 (concurrent recovery)
red pepper, fruit	0.1	0.1 0.5	76 76	74-78 76-77	2.1% 0.9%	3 3	< 0.01	external standard in controls	Cho, 2013, no code (concurrent recovery)
red pepper, leaves	0.1	0.1 0.5	71 72	71-72 72 (3x)	0.2% 0.2%	3 3	< 0.01	external standard in controls	Cho, 2013, no code (concurrent recovery)
soy bean, dried	0.01	0.01 0.5	92 94	89-94 92-95	2.1% 1.0%	6 6	< 0.01	external standard in untreated controls	JP2011C361 (concurrent recovery)
soy bean, green	0.01	0.01 0.5	77 89	72-78 88-92	3.0% 2.2%	6 6	< 0.01	external standard in untreated controls	JP2011C362 (concurrent recovery)
summer squash	0.01	 0.1	99 100	90-108 94-105	6.3% 3.8%	8 8	< 0.01	external standards in untreated controls	IB-2013-JAM-003-01-01 (concurrent recovery& method validation)
sweet pepper	0.01	0.01	93	78-102	9%	5	< 0.01	external std	JSM0337

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
		0.5	85	74-93	8%	5		in solvent 0.025-10 ng/mL linear	(procedural recovery)
sweet pepper	0.01	0.01 0.1 0.5	92 94 96	87-97 94-95 95-96	5% 1% 1%	3 3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0487 (procedural recovery)
sweet pepper	0.01	0.01 0.5	98 80	79-109 74-87	9% 5%	8 7	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0336 (procedural recovery)
sweet pepper	0.01	0.01 0.5	94 89	89-99 82-96	5% 7%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0485 (procedural recovery)
sweet pepper (bell)	0.01	0.01 0.1 0.2	105 na 100	100-115 104 94-106	6.7% na 5.7%	4 1 3	< 0.01	external standards in untreated controls	IB-2012-JLW-029-01-01 (concurrent recovery)
sweet pepper (non-bell)	0.01	0.01 0.1	100 101	92-104 94-105	5.0% 4.4%	5 5	< 0.01	external standards in untreated controls	IB-2012-JLW-029-01-01 (concurrent recovery)
tea, crude tea	0.02	0.02 2 5	90 89 91	87-92 84-92 84-99	2.4% 3.8% 7.6%	6 6 6	< 0.02	external std in solvent, linear, r=0.999	JP2011C133 (method recovery)
tea, infusion	0.02	0.02 2	88 92	86-91 91-93	2.3% 0.7%	6 6	< 0.02	external std in solvent, linear, r=0.999	JP2011C133 (method recovery)
tea, crude	0.02	0.2	84	79-93	9.0%	3	< 0.02	spiked control samples	JP2011C133 (quality control)
tea, infusion	0.02	0.2	88	89, 88	n.a.	2	< 0.02	spiked control samples	JP2011C133 (quality control)
tomato	0.01	0.01 0.5 1.0	95 86 na	75-109 76-92 82	11% 5% na	7 6 1	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0335 (procedural recovery)
tomato	0.01	0.01 0.5	101 90	87-110 88-91	9% 1%	5 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0486 (procedural recovery)
tomato	0.01	0.01 0.5	102 91	95-106 90-93	6% 2%	4 4	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0353 (procedural recovery)
tomato	0.01	0.01 0.5	88 91	74-97 87-99	10.5% 9.1%	6 6	< 0.01	spiked controls, linear, r=0.999	JP2012C105 (method recovery)
tomato	0.01	0.01 0.5	93 94	89-96 92-97	2.8% 2.1%	5 5	< 0.01	spiked samples, linear, r=0.999	JP2012C106 (method recovery)
tomato	0.01	0.01 0.5	74 101	72-77 98-103	3.1% 1.8%	6 6	< 0.01	spiked samples, linear, r=0.999	JP2011C132 (method recovery)
tomato	0.01	0.01 0.5	98 86	107, 90 84, 87	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0354 (procedural recovery)
tomato, canned	0.01	0.01 0.5	102 86	105, 98 86, 87	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0354 (procedural recovery)

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean range		RSD (%)	n	control mg/kg	calibration	Code no; Report no
tomato, (raw) juice	0.01	0.01 0.5	88 85	92, 83 83, 87	na na	2 2	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0354 (procedural recovery)
tomato	0.01	0.01	na	70 99	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, blanched	0.01	0.01	na	781	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, canned	0.01	1.0	na	93	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, peeled	0.01	0.01	na	119	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, dried	0.01	5	na	78	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, raw juice and juice	0.01	1	90	91, 89	na	2	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, ketchup, paste, puree	0.01	0.01 1 5	na 83	87 71, 95	na na	1 2	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, vegetable stock	0.01	0.01	na	100	na	1	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato, dry and wet pomace	0.01	5 15	96 90	91, 101 90, 91	na na	2 2	< 0.01	external standard in solvent 0.025-10 ng/mL linear, r= 0.9997	HH97BD (procedural recovery)
tomato	0.01	0.01 0.5	100 100	97-103 95-102	3% 4%	3 3	< 0.01	external std in solvent 0.025-10 ng/mL linear	JSM0488 (procedural recovery)
tomato	0.01	0.01 0.1	101 99 92	88-113 94 -105 92 - 92	7.3% 3.8% 0%	9 6 3	< 0.01	external standard, linear	IB-2012-JLW-029-01-01 (concurrent recovery)
tomato, puree	0.01	0.01 0.1	96 102	93-103 99-106	5.0% 2.8%	4 4	< 0.01	external standard, r= 0.9997	IB-2012-JLW-029-01-01 (method validation, including 1 concurrent recovery)
tomato paste	0.01	0.01 0.1	89 94	81-96 90-96	7.3% 3.0%	3 3	< 0.01	external standard, r= 1.000	IB-2012-JLW-029-01-01 (method validation, including 1 concurrent recovery)

Matrix	LOQ	Fortifi-cation level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
wheat, forage	0.01	0.01 0.5	90 106	85-95 104-109	4.8% 2.4%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, linear, r= 0.9994	120464 (ILV of method JSM0269)
wheat, forage	0.01	0.01 0.5	na na	92 81	na na	1 1	< 0.01	External std in solvent 0.025 – 10 ng/mL linear r=0.9985	JSM0414 (procedural recovery)
wheat, forage	0.01	0.01 0.1	89 94	76-97 88-100	8.4% 4.9%	6 6	< 0.01	External std in solvent 0.025 – 2.5 ng/mL linear r≥0.9994	IB-2012-JLW-022-01-01 (procedural recovery)
wheat, grain	0.01	0.01 0.5	96 106	93-103 104-108	4.6% 1.7%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, linear, r= 0.9996	120464 (ILV of method JSM0269)
wheat, grain	0.01	0.01 0.5	na na	99 110	na na	1 1	< 0.01	External std in solvent 0.025 – 10 ng/mL linear r=0.9985	JSM0414 (procedural recovery)
wheat, grain	0.01	0.01 0.1	89 92	80-101 85-100	8.1% 5.6	6 6	< 0.01	External std in solvent 0.025 – 2.5 ng/mL linear, r≥0.9994	IB-2012-JLW-022-01-01 (procedural recovery)
wheat, hay	0.01	0.01 0.25 0.5	na na na	108 97 94	na na na	1 1 1	< 0.01	External std in solvent 0.025 – 10 ng/mL linear r=0.9985	JSM0414 (procedural recovery)
wheat, straw	0.01	0.01 0.5	88 93	83-91 91-94	3.8% 1.7%	5 5	< 0.01	external standard 0.026 -10.4 ng/mL, linear, r= 0.9999	idem
wheat, straw	0.01	0.01 0.1 0.2	96 94 na	91-104 92-97 97	5.6% 2.7% na	6 5 1	< 0.01	External std in solvent 0.025 – 2.5 ng/mL linear, r≥0.9994	IB-2012-JLW-022-01-01 (procedural recovery)

nr=not reported; na = not applicable

[a] Data combined from peeled and apple without stems

LC-MS/MS Method XR44SB

An LC-MS/MS method [Miller, 2016a, report XR44SB] was developed and validated according to the full validation scheme for the determination degradation products BPQO, BCPBA and YT-1327 in tomato and grapes (RAC and processed fractions).

Extraction of the samples (20 gram solid or 20 mL liquid samples) follows the same procedures as in the other analytical methods; addition of standards where applicable, followed by two extractions with acetonitrile, combination of extracts, dilution with acetonitrile, and addition of 0.1% acetic acid. Extraction is followed by an HLB solid phase extraction (SPE) cleanup. Quantification was performed by LC-MS/MS monitoring two ion transitions to satisfy the confirmatory analysis requirement.

Quantitation ion transition: BPQO: *m/z* 343>308 (quantitation), BPQO: *m/z* 343>201 (confirmatory);

BCPBA*: m/z* 361>308 (quantitation), BCPBA: *m/z* 361>343 (confirmatory); YT-1327*: m/z* 258>190 (quantitation), YT-1327: *m/z* 258>173 (confirmatory).

The method was validated at 0.01 and 0.1 mg/kg for detection of BPQO, BCPBA and YT-1327 in tomato, tomato puree, grape and grape wine. The accuracy data were measured as analytical

recovery and precision data measured as relative stand deviation (RSD%) obtained for BPQO, BCPBA and YT-1327. These data using both quantitation and confirmatory ion transitions are summarised in Table 47. Final extract stability samples were fortified with 1 ng/mL. Stability samples analysed following initial fortification and after the storage period (0 and 5 days). Procedural recovery samples were fortified just prior to second analysis (following storage of stability samples). Procedural recoveries ranged from 102–108% in tomato, 94–101% in tomato puree, 84–98% in grape and 77–85% in grape wine (mean of two replicate samples). Matrix effects were assessed by comparing the chromatographic response of standards prepared in control final extract (Day 0 analysis) to standards prepared in solvent in the same manner.

This method was also used to determine these degradation products in processed commodities of grapes [Miller, 2016c, report BS38WY].

Table 47 Validation results for determining degradation products BPQO, BCPA and YT-1327 with LC-MS/MS method XR44SB in plant commodities

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean range		RSD (%)	n	control mg/kg	calibration	Code no; Report no
apple, without stems	BPQO	0.01	106	nr	-	1	< 0.01	external std in solvent 0.05-5 ng/mL Linear, r≥0.996	SQ74KP (procedural recovery)
apple, peeled	BPQO	0.01	105	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, peel	BPQO	1	89	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, core and stalks	BPQO	0.01	109	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, peeled, without core and stalks	BPQO	0.1	94	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, canned fruit	BPQO	0.1	94	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, stalks	BPQO	1	75	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, fruit syrup	BPQO	0.01	128	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, solid parts (after sieving)	BPQO	0.01	107	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple sauce, before pasteurization	BPQO	0.01	123	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple sauce, after pasteurization	BPQO	0.1	79	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, raw juice	BPQO	0.1	94	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, wet pomace	BPQO	0.1	86	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, dry pomace	BPQO	1	79	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, juice	BPQO	1	77	nr	-	1	< 0.01	idem	SQ74KP

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean range		RSD (%)	n	control mg/kg	calibration	Code no; Report no
									(procedural recovery)
apple, jelly	BPQO	1	77	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
grape	BPQO Quantitation (*m/z* 343>308)	0.01 0.1	78 95	73-84 82-102	5.7% 8.0%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9983	XR44SB
grape	BPQO Confirmation (*m/z* 343>201)	0.01 0.1	92 95	84-97 84-106	5.6% 9.0%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9994	XR44SB
grape, wine	BPQO Quantitation (*m/z* 343>308)	0.01 0.1	80 87	71-86 83-92	8.2% 4.4%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9983	XR44SB
grape, wine	BPQO Confirmation (*m/z* 343>201)	0.01 0.1	86 88	78-93 79-95	6.4% 6.5%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9994	XR44SB
grapes, bunches	BPQO	0.01 0.1	95 89	70-120 68-109	20.5% 21.0%	5 5	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
grape, must	BPQO	0.01 0.1	89 96	78-100	12.3%	3 1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
grape, wet pomace	BPQO	0.01 0.1	82 85	70-94 74, 96	14.6% na	4 2	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
grape, dry pomace	BPQO	0.01 0.1	na na	75 79	na na	1 1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
grape, juice	BPQO	0.01	na	98	na	1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
grape, wine	BPQO	0.01 0.1	na 83	93 77-89	na 5.5%	1 5	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
grape, raisins	BPQO	0.01 0.1	na 75	95 69-85	na 12	1 3	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9996	BS38WY (method recovery)
peaches, fruit w/o stones	BPQO	1	na	73	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9961	QK27SS (method recovery)
peaches, blanched/pureed/canned	BPQO	0.01 0.1 1	76 na na	70, 81 72 87	na na na	2 1 1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9961	QK27SS (method recovery)
peaches, peeled fruit	BPQO	0.01	na	95	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9961	QK27SS (method recovery)
peaches, (raw) juice	BPQO	0.01	98	80, 99	na	2	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9961	QK27SS (method recovery)
peaches, jam	BPQO	0.01	na	80	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9961	QK27SS (method recovery)
tomato	BPQO Quantitation (*m/z* 343>308)	0.01 0.1	85 88	74-95 74-97	9.7% 9.6%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9983	XR44SB
tomato	BPQO Confirmation (*m/z* 343>201)	0.01 0.1	84 85	71-97 76-93	11.5% 8.3%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9994	XR44SB

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
tomato puree	BPQO Quantitation (*m/z* 343>308)	0.01 0.1	80 80	75-86 76-84	5.9% 5.4%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9983	XR44SB
tomato puree	BPQO Confirmation (*m/z* 343>201)	0.01 0.1	86 82	78-91 77-86	6.4% 5.0%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9994	XR44SB
tomato, without stems	BPQO	0.01	na	95	na	1	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, blanched	BPQO	0.01	na	102	na	1	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, canned	BPQO	1.0	na	86	na	1	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, peeled	BPQO	0.01	na	69	na	1	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, dried	BPQO	5	na	95	na	1	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, raw juice and juice	BPQO	1	99	101, 97	na	2	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, ketchup, paste, puree	BPQO	0.01 1	na 84	92 77, 90	na na	1 2	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, vegetable stock	BPQO	0.01	na	97	na	1	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
tomato, dry and wet pomace	BPQO	5	104 107	101,	na	2	< 0.01	external standard in solvent 0.05-4 ng/mL linear r=0.9977	HH97BD (procedural recovery)
apple, without stems	BCPBA	0.01	120	nr	-	1	< 0.01	external std in solvent 0.05-5 ng/mL Linear, r≥0.996	SQ74KP (procedural recovery)
apple, peeled	BCPBA	0.01	109	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, peel	BCPBA	1	81	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, core and stalks	BCPBA	0.01	92	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, peeled, without core and stalks	BCPBA	0.1	69	nr	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, canned fruit	BCPBA	0.1	82	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
apple, stalks	BCPBA	1	81	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, fruit syrup	BCPBA	0.01	120	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, solid parts (after sieving)	BCPBA	0.01	98	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple sauce, before pasteurization	BCPBA	0.01	116	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple sauce, after pasteurization	BCPBA	0.1	70	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, raw juice	BCPBA	0.1	81	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, wet pomace	BCPBA	0.1	92	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, dry pomace	BCPBA	1	88	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, juice	BCPBA	1	78	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, jelly	BCPBA	1	83	na	-	1	< 0.01	idem	SQ74KP (procedural recovery)
grape	BCPBA Quantitation (*m/z* 361>308)	0.01 0.1	96 88	90-102 75-95	5.0% 9.9%	5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9999	XR44SB
grape	BCPBA Confirmation (*m/z* 361>343)	0.01 0.1	91 88	80-98 74-94	8.4% 9.5%	5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9996	XR44SB
grape, wine	BCPBA Quantitation (*m/z* 361>308)	0.01 0.1	86 92	83-94 89-93	5.1% 2.1%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9999	XR44SB
grape, wine	BCPBA Confirmation (*m/z* 361>343)	0.01 0.1	85 93	79-94 91-97	8.0% 2.9%	55	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9996	XR44SB
grapes, bunches	BCPBA	0.01 0.1	101 103	92-120 71-127	10.3% 21.1%	5 5	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9997	BS38WY (method recovery)
grape, must	BCPBA	0.01 0.1	90 88	84-100	10.0%	3 1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9997	BS38WY (method recovery)
grape, wet pomace	BCPBA	0.01 0.1	108 112	89-128 96, 127	15.3% na	4 2	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9997	BS38WY (method recovery)
grape, dry pomace	BCPBA	0.01 0.1	na na	102 88	na na	1 1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9997	BS38WY (method recovery)
grape, juice	BCPBA	0.01	na	102	na	1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9997	BS38WY (method recovery)
grape, wine	BCPBA	0.01 0.1	na 92	91 82-102	na 8.8%	1 5	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9997	BS38WY (method recovery)
grape, raisins	BCPBA	0.01	na	99	na	1	< 0.01	external standard	BS38WY

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
		0.1	97	83-116	17.4%	3		0.05 - 5 ng/mL, linear, r=0.9997	(method recovery)
peaches, fruit w/o stones	BCPBA	1	na	92	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9988	QK27SS (method recovery)
peaches, blanched/pureed/canned	BCPBA	0.01 0.1 1	80 na na	71, 89 86 92	na na na	2 1 1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9988	QK27SS (method recovery)
peaches, peeled fruit	BCPBA	0.01	na	78	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9988	QK27SS (method recovery)
peaches, (raw) juice	BCPBA	0.01	98	95, 102	na	2	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9988	QK27SS (method recovery)
peaches, jam	BCPBA	0.01	na	90	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9988	QK27SS (method recovery)
tomato	BCPA Quantitation (*m/z* 361>308)	0.01 0.1	91 92	85-97 86-94	4.8% 3.8%	5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9999	XR44SB
tomato	BCPBA Confirmation (*m/z* 361>343)	0.01 0.1	84 94	81-87 91-101	2.9% 4.2%	5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9996	XR44SB
tomato puree	BCPBA Quantitation (*m/z* 361>308)	0.01 0.1	88 93	81-93 92-96	5.3% 1.8%	5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9999	XR44SB
tomato puree	BCPBA Confirmation (*m/z* 361>343)	0.01 0.1	92 97	79-100 94-100	9.2% 3.1%	5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9996	XR44SB
tomato, without stems	BCPBA	0.01	na	110	na	1	< 0.01	external standard in solvent 0.05-5 ng/mL linear, r= 0.9993	HH97BD (procedural recovery)
tomato, blanched	BCPBA	0.01	na	114	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, canned	BCPBA	1.0	na	80	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, peeled	BCPBA	0.01	na	71	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, dried	BCPBA	5	na	112	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, raw juice and juice	BCPBA	1	116 121	111,	na	2	< 0.01	idem	HH97BD (procedural recovery)
tomato, ketchup, paste, puree	BCPBA	0.01 1	na 84	73 77, 98	na na	1 2	< 0.01	idem	HH97BD (procedural recovery)
tomato, vegetable stock	BCPBA	0.01	na	121	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, dry and wet pomace	BCPBA	5	87	95, 79	na	2	< 0.01	idem	HH97BD (procedural recovery)
apple, without stems	YT-1327	0.01	na	89	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, peeled	YT-1327	0.01	na	100	-	1	< 0.01	idem	SQ74KP

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean	range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
				nr					(procedural recovery)
apple, peel	YT-1327	1	na	80	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, core and stalks	YT-1327	0.01	na	99	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, peeled, without core and stalks	YT-1327	0.1	na	96	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, canned fruit	YT-1327	0.1	na	88	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, stalks	YT-1327	1	na	83	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, fruit syrup	YT-1327	0.01	na	112	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, solid parts (after sieving)	YT-1327	0.01	na	108	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple sauce, before pasteurization	YT-1327	0.01	na	109	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple sauce, after pasteurization	YT-1327	0.1	na	79	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, raw juice	YT-1327	0.1	na	94	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, wet pomace	YT-1327	0.1	na	94	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, dry pomace	YT-1327	1	na	94	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, juice	YT-1327	1	na	92	-	1	< 0.01	idem	SQ74KP (procedural recovery)
apple, jelly	YT-1327	1	na	88	-	1	< 0.01	idem	SQ74KP (procedural recovery)
grape	YT-1327 Quantitation (*m/z* 258>190)	0.01 0.1	102 106	99-113 97-111	6.0% 5.0%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9995	XR44SB
grape	YT-1327 Confirmation (*m/z* 258>173)	0.01 0.1	91 106 109	84-98 100-	6.3% 3.3%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9997	XR44SB
grape, wine	YT-1327 Quantitation (*m/z* 258>190)	0.01 0.1	79 93	72-83 90-97	5.4% 2.9%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9995	XR44SB
grape, wine	YT-1327 Confirmation (*m/z* 258>173)	0.01 0.1	83 93	77-89 91-96	5.2% 2.2%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9997	XR44SB
grapes, bunches	YT-1327	0.01 0.1	83 91	71-90 83-97	8.5% 7.0%	5 5	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9998	BS38WY (method recovery)
grape, must	YT-1327	0.01 0.1	76 na	70-79 96	6.5%	3 1	< 0.01	external standard 0.05 - 5 ng/mL,	BS38WY (method

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
							linear, r=0.9998	recovery)
grape, wet pomace	YT-1327	0.01 0.1	93 76-112 94 90, 99	20.1% na	4 2	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9998	BS38WY (method recovery)
grape, dry pomace	YT-1327	0.01 0.1	na 92 na 88	na na	1 1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9998	BS38WY (method recovery)
grape, juice	YT-1327	0.01	na 72	na	1	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9998	BS38WY (method recovery)
grape, wine	YT-1327	0.01 0.1	na 95 86 70-95	na 11.9%	1 5	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9998	BS38WY (method recovery)
grape, raisins	YT-1327	0.01 0.1	na 84 89 87-92	na 3%	1 3	< 0.01	external standard 0.05 - 5 ng/mL, linear, r=0.9998	BS38WY (method recovery)
peaches, fruit w/o stones	BCPBA	1	na 92	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9988	QK27SS (method recovery)
peaches, fruit w/o stones	YT-1327	1	na 97	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9971	QK27SS (method recovery)
peaches, blanched/pureed/canned	YT-1327	0.01 0.1 1	82 72, 92 na 91 na 85	na na na	2 1 1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9971	QK27SS (method recovery)
peaches, peeled fruit	YT-1327	0.01	na 110	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9971	QK27SS (method recovery)
peaches, (raw) juice	YT-1327	0.01	96 94, 98	na	2	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9971	QK27SS (method recovery)
peaches, jam	YT-1327	0.01	na 83	na	1	< 0.01	external standard 0.05 to 5 ng/mL, linear, r=0.9971	QK27SS (method recovery)
tomato	YT-1327 Quantitation (*m/z* 258>190)	0.01 0.1	92 87-98 94 87-99	4.9% 5.5%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9995	XR44SB
tomato	YT-1327 Confirmation (*m/z* 258>173)	0.01 0.1	91 85-102 96 90-102	7.0% 5.0%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9997	XR44SB
tomato puree	YT-1327 Quantitation (*m/z* 258>190)	0.01 0.1	83 68-93 93 85-100	11.1% 7.2%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r= 0.9995	XR44SB
tomato puree	YT-1327 Confirmation (*m/z* 258>173)	0.01 0.1	88 74-96 96 90-101	10.0% 5.1%	5 5	< 0.01 (n=2)	external standard 0.05 - 5 ng/mL, linear, r=0.9997	XR44SB
tomato, without stems	YT-1327	0.01	na 101	na	1	< 0.01	external standard in solvent 0.05-5 ng/mL linear r=0.9987	HH97BD (procedural recovery)
tomato, blanched	YT-1327	0.01	na 88	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, canned	YT-1327	1.0	na 91	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, peeled	YT-1327	0.01	na 82	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, dried	YT-1327	5	na 100	na	1	< 0.01	idem	HH97BD (procedural

Matrix	Analyte (reported LOQs < 0.01 mg/kg)	Fortification level (mg/kg)	Recovery (%) Mean range	RSD (%)	n	control mg/kg	calibration	Code no; Report no
								recovery)
tomato, raw juice and juice	YT-1327	1	81 68, 94	na	2	< 0.01	idem	HH97BD (procedural recovery)
tomato, ketchup, paste, puree	YT-1327	0.01 1	na 99 86 84, 88	na na	1 2	< 0.01	idem	HH97BD (procedural recovery)
tomato, vegetable stock	YT-1327	0.01	na 97	na	1	< 0.01	idem	HH97BD (procedural recovery)
tomato, dry and wet pomace	YT-1327	5	97 98, 96	na	2	< 0.01	idem	HH97BD (procedural recovery)

Analytical methods used in study reports in animal commodities

Analytical method used in the dietary feeding study [Ross, 2013, JSM0515] was LC-MS/MS method JSM0277, described under enforcement methods.

Stability of pesticide residues in stored analytical samples

The Meeting received information on storage stability of parent and NK-1375 in raw plant commodities and of parent, NK-1375, NSY-27, NSY-28 and YT-1284 in animal commodities. One study specific storage stability study of raw plant commodities was submitted (study 1 below). In addition, storage stability data are included in the various field residue trials, processing studies and animal metabolism and feeding studies. The results are summarised in this section.

Storage stability in animal matrices

In the goat metabolism study [Kane, 2013, report JSM0059] tissues were first analysed within 2 months after sacrifice. The lasts analyses were performed within 125 days after sacrifice. Reanalysis approximately 2 months after the last analysis was performed. Raw data were not provided, but the report stated that the results confirm the stability during storage. Though small quantitative changes had occurred, the metabolic profile remained unchanged. Table 48 provides an overview of the additional data, not included in the primary report, to show the storage stability within study JSM 0059. The trial did not include storage stability data from day 0.

Table 48 Storage stability of 0.10 mg/kg parent, YT-1284 and NSY-27 in animal matrices stored frozen at -20 °C (phenyl label and pyrazole label)

Matrix	parent mg/kg	parent mg/kg + 2 months	parent % recovery	YT-1284 mg/kg	YT-1284 + 2 months	YT-1284 % recovery	NSY-28 mg/kg	NSY-28 mg/kg + 2 months	NSY-28 % recovery
		phenyl							
liver	31.6	32.9	104	12.1	12.5	103	24.2	32.2	133
kidney	28.3	31.8	112	10.5	6.7	64	34.7	40.3	116
muscle	43.8	49.6	113	16.9	10.8	64	27.0	30.6	113
fat	76.4	80.6	105	6.0	0.6	10	8.0	7.6	95
whole milk	71.4	86.2	121	11.2	3.1	28	5.2	2.7	52
		pyrazole label							
liver	30.1	31.8	106	16.9	16.4	97	31.4	31.8	101
kidney	19.0	17.6	93	10.7	8.8	82	53.2	61.2	115
muscle	22.7	24.4	107	17.3	14.5	84	45.9	51.5	112
fat	44.3	44.6	101	5.7	0.4	7.0	43.4	52.4	121
whole	58.4	67.7	116	21.2	13.6	64	9.4	5.4	57

Matrix	parent mg/kg	parent mg/kg + 2 months	parent % recovery	YT-1284 mg/kg	YT-1284 + 2 months	YT-1284 % recovery	NSY-28 mg/kg	NSY-28 mg/kg + 2 months	NSY-28 % recovery
		phenyl							
milk									

In the hen metabolism study [Jones, 2013, report JSM0060] tissues and eggs were first analysed within 2 months after sacrifice with the last analysis being performed within a maximum of 155 days. Reanalyses of the samples of liver, muscle, fat, skin and egg was performed within 2 months after the last analyses. The chromatograms showed that no major changes had occurred in the proportions of components during storage of extracts for periods of approximately six months under frozen conditions (<-18 ºC). The trial did not include storage stability data from day 0.

In a residue transfer study (feeding study) in dairy cows [Ross, A, 2013, report JSM0515] tissues were analysed on day 0 and again 39 days later by spiking untreated samples of tissues. The results are summarised in Table 49 and Table 50. The results provided are the means of two replicate samples.

Table 49 Storage stability of 0.10 mg/kg parent, NK-1375 and NSY-27 in liver, kidney, muscle and fat stored at -20 °C

Matrix	parent mg/kg day 0	parent mg/kg day 39	parent % recovery	NK-1375 mg/kg day 0	NK1375 mg/kg day 39	NK-1375 % recovery	NSY-27 mg/kg day 0	NSY-27 mg/kg day 39	NSY-27 % recovery
liver	0.078	0.090	115	0.082	0.097	118	0.076	0.083	109
kidney	0.082	0.086	105	0.105	0.077	73	0.088	0.105	119
muscle	0.072	0.066	92	0.095	0.063	66	0.091	0.063	69
fat	0.084	0.094	112	0.100	0.076	76	0.101	0.091	90

Table 50 Storage stability of 0.10 mg/kg NSY-28 and YT-1284 in liver, kidney, muscle and fat stored at -20 °C

Matrix	NSY-28 mg/kg day 0	NSY-28 mg/kg day 39	NSY-28 % recovery	YT-1284 mg/kg day 0	YT-1284 mg/kg day 39	YT-1284 % recovery
liver	0.084	0.085	101	0.079	0.088	111
kidney	0.089	0.083	93	0.094	0.103	110
muscle	0.091	0.061	67	0.080	0.088	110
fat	0.083	0.074	89	0.095	0.104	109

The storage stability data form the two animal metabolism studies do not cover the initial storage period of 0 days to 2 months. The storage period between sacrifice and first analysis is only two months. Follow-up analysis in these studies show similar results. Combning the findings storage stability findings in the metabolism studies with the 39 day storage stability study (days 0–39) included in the dairy feeding study suggests that the storage stability is sufficiently covered for the animal matrices in the evaluated animal studies.

Storage stability in plant matrices

Study 1

Storage stability was investigated by spiking various homogenised plant commodities (wine, canola, grapes, lettuce, potatoes, broccoli, and dry beans) with 0.1 mg/kg parent or NK-1375 (one analyte per sample) [Miller, 2014, report JSM0423]. Samples were stored for 1–18 months at -20 ºC and were analysed in duplicate at various intervals. LC-MS/MS method JSM0269 was used for quantification of parent and metabolite in all crops. The method is considered valid for the purpose of this study (commodity type and concentration level of the analytes). Average concurrent recoveries were within

70–120% for each analyte and matrix and control samples had residues < 0.0025 mg/kg, showing adequate performance at the time of analysis of the samples.

Storage stability results (not corrected for concurrent recovery) and concurrent recoveries are shown in Table 51. Residues of parent and NK-1375 are stable for at least 18 months in crop commodities representative of the high water (lettuce, broccoli), high acid (grapes), high starch (potato), high protein (dry beans), and high oil (canola–oilseed rape seeds) commodity groups as well as in wine when stored at or below -20 °C.

Table 51 Storage stability of 0.10 mg/kg parent or NK-1375 in crop commodities stored at -20 °C

Matrix	Storage period (months)	parent mg/kg[a]	parent Mean % remaining[b]	parent Mean concurrent recovery (%)	NK-1375 mg/kg[a]	NK1375 Mean % remaining[b]	NK-1375 Mean concurrent recovery (%)
wine	0	0.099	100*	nr	0.095	100*	nr
	1	0.092	93	95	0.088	93	96
	3	0.081	82	94	0.082	86	96
	6	0.081	82	81	0.075	79	82
	12	0.100	101	105	0.074	78	95
	18	0.099	100	103	0.087	92	104
canola (oilseed rape seeds)	0	0.092	100*	nr	0.092	100*	nr
	1	0.081	88	83	0.076	83	84
	3	0.072	78	88	0.081	88	91
	6	0.074	80	75	0.075	82	82
	12	0.089	97	103	0.074	80	92
	18	0.081	88	96	0.079	86	91
grapes	0	0.097	100*	nr	0.094	100*	nr
	1	0.082	85	92	0.082	87	90
	3	0.078	80	95	0.085	90	94
	6	0.080	82	80	0.080	85	81
	12	0.088	91	95	0.083	88	96
	18	0.077	79	97	0.086	91	107
lettuce	0	0.092	100*	nr	0.091	100*	nr
	1	0.078	85	85	0.081	89	81
	3	0.084	91	90	0.086	95	90
	6	0.080	87	77	0.075	82	74
	12	0.091	99	94	0.079	87	83
	18	0.082	89	90	0.085	93	86
potato	0	0.097	100*	nr	0.107	100*	nr
	1	0.088	91	91	0.094	88	92
	3	0.083	86	89	0.090	84	94
	6	0.082	85	75	0.088	82	86
	12	0.098	101	94	0.088	82	95
	18	0.098	101	99	0.109	102	110
broccoli	0	0.090	100*	nr	0.087	100*	nr
	1	0.077	86	80	0.075	86	80
	3	0.078	87	86	0.080	92	90
	6	0.075	83	72	0.073	84	80
	12	0.084	93	90	0.073	84	88
	18	0.072	80	86	0.075	86	85
dry beans	0	0.089	100*	nr	0.087	100*	nr
	1	0.076	85	82	0.077	89	81
	3	0.081	91	85	0.082	94	89
	6	0.075	84	73	0.072	83	72
	12	0.087	98	87	0.078	90	80
	18	0.078	88	78	0.078	90	88

[a] Results as presented in the study report; corrected for corresponding procedural recovery amount (mg/kg detected × procedural recovery).

[b] Results not corrected for concurrent recovery.

* The % remaining was set at 100% at time zero. If another level was found at time zero, instead of the expected 0.10 mg/kg, that level was set at 100%, to see the development of this residue level in time.

In addition to the specific storage stability study a number of field residue trials storage stability samples were tested. The storage stability results are presented in Table 52 and Table 53. Samples were reported as uncorrected for average concurrent method recoveries.

Table 52 Storage stability of cyclaniliprole in various crops as determined in field residue trials

Commodity	Storage time (days)	Spike level (mg/kg)	% remaining	Reference
Cherries	85 93	0.5 0.5	80 (n=2) 82 (n=2)	Kuaike & Naruto, 2013, no code
Chinese cabbage	12 24	0.5 0.5	96 (n=2) 98 (n=2)	Yoshiyuki, 2013c, report JP2012C108
Chinese cabbage	11 15 33 40	0.5 0.5 0.5 0.5	92 (n=2) 93 (n=2) 95 (n=2) 95 (n=2)	Hitoshi, 2013, report JP2013C091
grapes	36 89 74	0.5 0.5 0.5	93 (n=2) 94 (n=2) 98 (n=2)	Kimikazu, 2013a, report JP2012C100
grapes	31	0.5	82 (n=2)	Kimikazu, 2013b, report JP2013C280
red pepper, fruit	131	0.5	83-84 (n=3)	Cho, 2013, no code
red pepper, leaf	131	2.5	74-75 (n=3)	Cho, 2013, no code
tea	173	2	87-90% (n=4)	Koki, 2012, report JP2012C133
tea	114-181	2	88-99% (n=4)	Yoshiyuki, 2013f, report JP2013C101
tomato	44-253	0.5	98-101 (n=6)	Yoshiyuki, 2013a, report JP2012C105
tomato	127	0.5	100 (n=2)	Yoshiyuki, 2013b, report JP2012C106
tomato	161	0.5	88-97 (n=4)	Kouji, 2012, report JP2011C132
soy bean, dried	189 152	0.5 0.5	98 (n=2) 95 (n=2)	Takashi, 2012b, report JP2011C361
soy bean, dried	76-109	0.5	96-99% (n=8)	Yoshiyuki, 2013d, report JP2012C102
soy bean, green	220 227	0.5 0.5	84 (n=2) 86 (n=2)	Takashi, 2012a, report JP2011C362
soy bean, green	105	0.5	74 (n=2)	Yoshiyuki, 2013d, report JP2012C103

Table 53 Storage stability of metabolite NK-1375 in various crops as determined in field residue trials

Commodity	Storage time (days)	Spike level (mg/kg)	% remaining	Reference
Cherries	85 93	0.5 0.5	80 (n=2) 83 (n=2)	Kuzaki & Naruto, 2013, no code
Chinese cabbage	12 24	0.5 0.5	90 (n=2) 98 (n=2)	Yoshiyuki, 2013c, report JP2012C108
Chinese cabbage	11 15 33 40	0.5 0.5 0.5 0.5	91 (n=2) 95 (n=2) 91 (n=2) 91 (n=2)	Hitoshi, 2013, report JP2013C091
grapes	36 89 74	0.5 0.5 0.5	96 (n=2) 94 (n=2) 94 (n=2)	Kimikazu, 2013a, report JP2012C100
grapes	31	0.5	88 (n=2)	Kimikazu, 2013b, report JP2013C280
red pepper, fruit	131	0.5	73-75 (n=3)	Cho, 2013, no code
red pepper, leaf	131	2.5	71-72 (n=3)	Cho, 2013, no code
tea	173	2	84-92% (n=4)	Koki, 2012, report JP2012C133
tea	114-181	2	77-86% (n=4)	Yoshiyuki, 2013f, report JP2013C101
tomato	44-253	0.5	86-94% (n=6)	Yoshiyuki, 2013a, report JP2012C105
tomato	127	0.5	93, 98 (n=2)	Yoshiyuki, 2013b, report JP2012C106
tomato	161	0.5	92-94 (n=4)	Kouji, 2012, report JP2011C132

Commodity	Storage time (days)	Spike level (mg/kg)	% remaining	Reference
soya bean, dried	189 152	0.5 0.5	94 (n=2) 94 (n=2)	Takashi, 2012b, report JP2011C361
soya bean, dried	76-109	0.5	86-90% (n=8)	Yoshiyuki, 2013d, report JP2012C102
soya bean, green	220 227	0.5 0.5	90 (n=2) 82 (n=2)	Takashi, 2012a, report JP2011C362
soya bean, green	105	0.5	78 (n=2)	Yoshiyuki, 2013d, report JP2012C103

USE PATTERN

The insecticide cyclaniliprole is used as an insecticide in a wide range of crops. Cyclaniliprole is an anthranilic diamide with the mode/target of action being Ryanodine receptor modulation in insects. A registered label for cyclaniliprole containing products has been submitted for uses in Korea for Lapitan, Cyclaniliprole SL, containing 4.5% cyclaniliprole and in the USA for CYCLANILIPROLE 50 SL INSECTICIDE, also containing 4.5% cyclaniliprole. The product label refers to a broader range of crops, which are listed below Table 54. It is noted that the crop grouping as indicated in the labels does not necessarily correspond with the grouping of the Codex, e.g. the USA label for stone fruits should be three crop groups: cherries (003A); plums (003B), and peaches, nectarines, and apricots (003C). The same applies for the brassica's and the leafy vegetables: kale and mustard greens belong to the leafy vegetables instead of the brassicas.

Table 54 Registered pre-harvest uses of cyclaniliprole

Crop	Country	Form	Application				PHI, days
			Method	Rate g ai/ha	Spray conc, g ai/hL	Number (interval in days)	
Pome fruits[a] - apple, - pear	USA	Cyclaniliprole 50SL	foliar spray	60-80 (seasonal max 300)	2.1-8.6[b]	3-5[c] (10)	7[d]
Stone fruits[e] - cherries, - peaches, - plums, - apricots, and - nectarines	USA	Cyclaniliprole 50SL	foliar spray	60-80 (seasonal max 300)	2.1-8.6[b]	3-5[c] (7)	7[d]
Small fruit (vine climbing fruit)[f] - grapes	USA	Cyclaniliprole 50SL	foliar spray	60-80 (seasonal max 300)	4.3-8.6[b]	3-5[c] (7)	7[d]
Brassica's[g] - broccoli - Brussels sprouts - cabbage - cauliflower - kale - mustard greens	USA	Cyclaniliprole 50SL	foliar spray	40-60 (seasonal max 240)	2.1-6.4[b]	4-6 [h] (5)	1[d]
Chinese cabbage	Republic of Korea	SL	foliar application	45	2.2	2 (10)	14
Cucurbit vegetables[i] - cucumber - muskmelon - summer squash	USA	Cyclaniliprole 50SL	foliar spray	40-60 (seasonal max 240)	2.1-6.4[b]	4-6[h] (5)	1[d]
Fruiting vegetables[j] - tomato - bell pepper - nonbell pepper	USA	Cyclaniliprole 50SL	foliar spray	40-60 (seasonal max 240)	2.1-6.4[b]	4-6[h] (5)	1[d]
Chili pepper	Republic of Korea	SL	foliar application	45	2.2	2[x] (10)	3

Crop	Country	Form	Application				PHI, days
Leafy vegetables (non-brassica)[k] - head lettuce - leaf lettuce - spinach	USA	Cyclaniliprole 50SL	foliar spray	40-60 (seasonal max 240)	2.1-6.4[b]	4-6[i] (5)	1[d]
Leek (Welsh onion)	Republic of Korea	SL	foliar application	45	2.2	2 (10)	3
Tree nuts[l] - almond, - pecan	USA	Cyclaniliprole 50SL	foliar spray	60-80 (seasonal max 300)	2.1-8.6[b]	3-5[d] (10)	30[d]

[x] In primary submission this was 3 times. However, translated registered label clearly states 2 times

[a] According to the submitted label this includes all members of the Pome Crop Group Family: apple; azarole; crabapple; loquat; mayhaw; medlar; pear; pear, Asian; quince; quince, Chinese; quince, Japanese; tejocote; cultivars, varieties and/or hybrids of these.

[b] Spray volumes will usually range from 187-935 L/ha for diluate sprays and ca. 47-94 L/ha for concentrated ground and aerial sprays. Best results: pome fruit, stone fruits, tree nuts: 935-1869 L/ha; small fruit vine climbing: 935-1400 L/ha.

[c] Do not apply more than 0.3 kg ai/ha per year.

[d] Plant back interval 30 days for not registered crops.

[e] According to the submitted label this includes all members of the Stone Fruit Crop Group Family including apricot; apricot, Japanese; capulin; cherry, black; cherry, Nanking; cherry, sweet; cherry, tart; Jujube, Chinese; nectarine; peach; plum; plum, American; plum, beach; plum, Canada; plum, cherry; plum, Chickasaw; plum, Damson; plum, Japanese; plum, Klamath; plum, prune; plumcot; sloe; cultivars, varieties, and/or hybrids of these.

[f] According to the submitted label (not registered) this includes all members of the Small Fruit Vine Climbing Crop Subgroup 13-07F, except fuzzy kiwifruit including Amur river grape; gooseberry; grape; kiwifruit, hardy; maypop; schisandra berry; cultivars, varieties, and/or hybrids of these.

[g] According to the submitted label this includes all members of the Brassica Vegetable Crop Group Family including broccoli; broccoli Chinese (gai lon); broccoli raab (rapini); Brussels sprouts; cabbage; Chinese cabbage (bok choy); cabbage (napa); Chinese mustard (gai choy); cauliflower; cavalo broccoli; collards; kale; kohlrabi; mizuna; mustard greens; mustard spinach; rape greens.

[h] Do not apply more than 0.24 kg ai/ha per year (for brassica and non brassica leafy vegetables: max 6 × 0.04 kg ai/ha/year or max 4 × 0.06 kg ai/ha/year). For resistence management do not apply more than 3 times within a single generation of insect pest(s) on a crop.

[i] According to the submitted label this includes all members of the Cucurbit Vegetable Crop Group Family including Chayote (fruit); Chinese waxgourd (Chinese preserving melon); Citron melon; Cucumber; Gherkin; Gourd, edible (includes hyotan, cucuzza, hechima, Chinese okra); *Momordica spp* (includes balsam apple, balsam pear, bitter melon, Chinese cucumber); Muskmelon, hybrids and/or cultivars of *Cucumis melo* (includes true cantaloupe, cantaloupe, casaba, crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon, and snake melon); Pumpkin; Squash, summer (includes crookneck squash, scallop squash, straightneck squash, vegetable marrow, zucchini); Squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash; Watermelon (includes hybrids and/or varieties of *Citrullus lanatus*).

[j] According to the submitted label this includes all members of the Fruiting Vegetable Crop Group Family including African eggplant; bush tomato; bell pepper; cocona; currant tomato; eggplant; garden huckleberry; goji berry; groundcherry; martynia; naranjilla; okra; pea eggplant; pepino; nonbell pepper; roselle; scarlet eggplant; sunberry; tomatillo; tomato; tree tomato; cultivars, varieties, and/or hybrids of these.

[k] According to the submitted label this includes all members of the Leafy Vegetable Crop Group Family including Amaranth, leafy; arugula (roqette); cardoon; celtuce; chervil; Chinese spinach; chrysanthemum (edible leaved); chrysanthemum (garland); corn salad cress (garden); dandelion; dock (sorrel); endive (escarole); Florence fennel; lettuce (head and leaf); orach; parsley; purslane (garden and winter); radicchio (red chicory); rhubarb; spinach; spinach, vine; spinach, New Zealand; Swiss chard

[l] According to the submitted label this includes all members of the Tree Nut Crop Group Family including African nut-tree; Almond; Beechnut; Brazil nut; Brazilian pine; Bunya; Bur oak; Butternut; Cajou nut; Candlenut; Cashew; Chestnut; Chinquapin; Coconut; Coquito nut; Dika nut; Ginkgo; Guiana chestnut; Hazelnut; Heartnut; Hickory nut; Japanese horse-chestnut; Macadamia nut; Mongongo nut; Monkey-pot; Monkey puzzle nut; Okari nut; Pachira nut; Peach palm nut; Pecan; Pequi; Pili nut; Pine nut; Pistachio; Sapucaia nut; Tropical almond; Walnut, black; Walnut, English; Yellowhorn; cultivars, varieties and/or hybrids of these.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised residue trials for the following crops for foliar spray applications:

Foliar spray applications

Group/Sub-group	Commodity	Table No.
Pome fruits	Apples	55, 56 & 57
	Pears	58
Stone fruits		
Cherry,	Cherries	59 & 60
Plum,	Plums	61 & 62
Apricots, peach, nectarine	Apricot	63
	Peaches	64 & 65
Berries and small fruits		
Small fruit and vine climbing	Table grapes	66

Group/Sub-group	Commodity	Table No.
	Wine grapes	67
	Table and wine grape	68 & 69
Brassicas	Broccoli	70 & 71
	Cauliflower	72
	Head cabbages	73, 74
	Brussels sprouts	75
Fruiting vegetables - cucurbits	Cucumbers	76
	Summer squash	77
	Melons	78
Fruiting vegetables, other than cucurbits	Peppers (field)	79, 80 & 81
	Peppers (indoor)	82
	Peppers, Chili	83
	Tomatoes (field)	84, 85 & 86
	Tomatoes (indoor)	87
Leafy vegetables (including Brassica leafy vegetables)		
	Lettuce (head)	88
	Lettuce (leaf)	89
	Spinach	90
	Chinese cabbage	91
	Kale	92
	Mustard greens	93
Legume vegetables	Soya beans (green + pods)	94
Pulses	Soya beans, dry	95
Tree nuts	Almond & Pecan	96
Infusions	Tea	97
Miscellaneous fodder and forage:	Almond hulls	98

Application rates, spray concentrations and residues have been rounded to two figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless otherwise stated. Unquantifiable residues are shown as below the reported LOQ (e.g. < 0.01 mg/kg). Where multiple analyses were conducted on a single sample, the average value is reported. Where multiple samples were taken from a single plot, the individual and average values are reported. Where results from separate plots with distinguishing characteristics such as different formulations, crop varieties or treatment schedules were reported, results are listed separately for each plot. Residues from the trials conducted according to the critical GAP, which has been used for the estimation of maximum residue levels, STMR and HR values are underlined.

The residues presented in the tables are given as cyclaniliprole and metabolite NK-1375, expressed as themselves. The total residues (sum of the mean of parent and NK-1375) are expressed as parent equivalents by applying a conversion factor of 1.064 to NK-1375. Levels of NK-1375 are generally not detectable if parent concentrations are < 0.01 mg/kg. Field trials resulting in significant residue levels show a maximum contribution of NK-1375 of approximately 30% to the total residue. Therefore, if both parent and metabolite are < 0.01 mg/kg, the total is calculated as < 0.01 mg/kg.

In some trials either single field trials or duplicate or even triplicate field samples were taken at various time points. Results are therefore sometimes presented as single values or as duplicate/triplicate single values with the (mean) value between brackets.

The field and analytical data were generally sufficiently described. Samples marked with "[GS]' indicate that the samples were not of commercial standards, because the harvest was too early. Sample sizes were in accordance with the FAO manual 2016 appendix V, except when marked by "[SS]" in the table. Results marked with "[GS]" or "[SS]" are not selected for derivation of the MRL, if according to cGAP.

In the tables the following abbreviations were used:

- na = not analysed

- ns = not stated, not reported, not specified
- Form = formulations: SL = soluble concentrate;
- ADJ = adjuvant; NIS = non ionic surfactant as adjuvant; COC = crop oil concentrate as adjuvant
- GS = growth stage at last application
- DALT = days after last application

Pome fruit

Apples

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in field residue trials on apples in the USA (15) and in Canada (1) [Wiedmann and McDonald, 2013b, report IB-2012-JLW-020, Wiedmann and McDonald, 2014a, report IB-2013-JLW-004]. Three spray applications at 99–158 g ai/ha, with an interval of 13–15 days were applied with spray volumes ranging from 944–1998 L/ha. Trials were performed in the growing seasons 2012–2013 and include two decline trials. The results are summarised in Table 55.

Table 55 Supervised field trials on apples (fruit) treated with a pre-harvest foliar application with a formulation of cyclaniliprole (50SL)

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
North Rose, NY, USA, 2012 (Rome)	3 (14)	101 100100	9 9 9	BBCH 85 Sept 21	7	0.059/0.077 (0.068)	< 0.01/< 0.01(< 0.01)	0.070/ 0.089 (0.079)	IB-2012-JLW-020; IB-2012-JLW-020-01 [Wiedmann & McDonald, 2013b]
idem	3 (14)	995 1010 999	85 85 85	idem	7	0.71[a]	0.078 [a]	0.79	idem, see processing
Hereford, PA, USA, 2012 (Red delicious)	3 (14-15)	101100101	5 5 5	BBCH 87 Sept 07	0 3 6 10	0.11/0.13 (0.12) 0.073/0.064 (0.069) 0.047/0.061 (0.054) 0.052/0.045 (0.049)	< 0.01/< 0.01(< 0.01) < 0.01/< 0.01(< 0.01) < 0.01/< 0.01(< 0.01) < 0.01/< 0.01(< 0.01)	0.12/ 0.14 (0.13) 0.084/ 0.075 (0.079) 0.058/ 0.072 (0.065) 0.063/ 0.056 (0.059)	IB-2012-JLW-020; IB-2012-JLW-020-02 [Wiedmann & McDonald, 2013b]
Blairsville, GA, USA, 2012 (Rome)	3 (14)	102 104 103	10 10 10	BBCH 87 Aug 06	7	0.035/0.035(0.035)	< 0.01/< 0.01(< 0.01)	0.046/ 0.046 (0.046)	IB-2012-JLW-020; IB-2012-JLW-020-03 [Wiedmann & McDonald, 2013b]
Deerfield, MI, USA, 2012 (Red delicious)	3 (14)	153 158 150	9 9 9	BBCH 87 Sept 05	7	0.016[b]	< 0.01[b]	0.027	IB-2012-JLW-020; IB-2012-JLW-020-04 [Wiedmann &

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg Parent	NK-1375	Tot.	Report; Trial no [ref]
									McDonald, 2013b]
idem	idem	151153 150	8 8 8	idem	7	0.010[b]	< 0.01[b]	0.021	idem
Oregon, WI, USA, 2012 (Cortland)	3 (14)	100100100	9 8 9	5.1-7.6 cm fruit diameter Sept 12	7	0.022/0.023(0.023)	< 0.01/< 0.01(< 0.01)	0.033/ 0.034 (0.033)	IB-2012-JLW-020-01-01; IB-2012-JLW-020-05 [Wiedmann & McDonald, 2013b]
Perry, UT, USA, 2012 (Gala) + [ADJ]	3 (13-15)	103 101 102	5 5 5	BBCH 85 Aug 21	7	0.059/0.050 (0.055)	0.012/0.01 (0.012)	0.072/ 0.062 (0.067)	IB-2012-JLW-020-; IB-2012-JLW-020-07 [Wiedmann & McDonald, 2013b]
Los Molinos, CA, USA, 2012 (Summerfield)	3 (14)	99 99 99	7 7 7	BBCH 81 Aug 23	7	0.032/0.042 (0.037)	0.012/0.019(0.016)	0.045/ 0.062 (0.053)	IB-2012-JLW-020-01-01; IB-2012-JLW-020-08 [Wiedmann & McDonald, 2013b]
Ephrata, WA, USA 2012 (Gala)	3 (14)	100101 99	5 5 5	BBCH 85 Aug 23	7	0.13/0.14 (0.13)	0.035/0.031(0.033)	0.17/ 0.17 (0.17)	IB-2012-JLW-020; IB-2012-JLW-020-09 [Wiedmann & McDonald, 2013b]
Payette, ID, USA, 2012 (Early Spur Rome)	3 (14-15)	100 101 101	11 11 11	fruit with advanced colour Sept 27	0 3 7 10	0.13/0.14 (0.14) 0.13/0.13 (0.13) 0.11/0.092 (0.10) 0.095/0.079 (0.087)	0.010/0.012 (0.011) 0.013/0.014 (0.014) 0.013/0.016 (0.015) 0.013/< 0.01 (0.012)	0.14/ 0.16 (0.15) 0.14/ 0.14 (0.14) 0.12/ 0.11 (0.12) 0.11/ 0.09 (0.10)	IB-2012-JLW-020; IB-2012-JLW-020-10 [Wiedmann & McDonald, 2013b]
North Rose, NY, USA, 2013 (Ida Red)	3 (14)	100 100 100	8 9 9	BBCH 85 Sept 10	7	0.050/0.041(0.046)	< 0.01/< 0.01(< 0.01)	0.061/ 0.052 (0.056)	IB-2013-JLW-004; IB-2013-JLW-004-01 [Wiedmann and McDonald, 2014a]
Conklin, MI, USA, 2013 (Ida Red)	3 (14)	100 100 100	8 9 9	BBCH 85 Sept 24	7	0.048/0.049 (0.049)	< 0.01/< 0.01(< 0.01)	0.059/ 0.060 (0.059)	IB-2013-JLW-004; IB-2013-JLW-004-02 [Wiedmann and McDonald, 2014a]
Wyoming, IL, USA,	3 (14)	101 102	7 7	90% mature	8	0.098/0.111(0.10)	0.026/0.019(0.023)	0.13/ 0.13	IB-2013-JLW-004;

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2013 (Gala)		101	7	Aug 26				(0.13)	IB-2013-JLW-004-03 [Wiedmann and McDonald, 2014a]
Oregon, WI, USA, 2013 (Paula Red)	3 (14)	99 101 100	9 9 9	5.7-7.0 cm fruit Aug 15	7	0.011/0.014 (0.013)	< 0.01/< 0.01(< 0.01)	0.022/ 0.025 (0.023)	IB-2013-JLW-004; IB-2013-JLW-004-04 [Wiedmann and McDonald, 2014a]
Ephrata, WA, USA, 2013 (Gala)	3 (14)	100 100 100	8 9 9	BBCH 85 Aug 22	7	0.063/0.053 (0.058)	0.015/0.013(0.014)	0.079/ 0.067 (0.073)	IB-2013-JLW-004; IB-2013-JLW-004-06 [Wiedmann and McDonald, 2014a]
Payette, ID, USA, 2013 (Early Spur Rome) + [ADJ]	3 (13, 15)	101 102103	7 7 7	advanced fruit colouring Sept 26	7	0.058/0.050 (0.054)	< 0.01/< 0.01(< 0.01)	0.069/ 0.061 (0.065)	IB-2013-JLW-004; IB-2013-JLW-004-07 [Wiedmann and McDonald, 2014a]
Simcoe, Ontario, Canada, 2012 (Golden Delicious)	3 (14)	106 101101	11 11 11	BBCH 85-87 Sept 27	7	0.066/0.070(0.068)	0.015/0.015 (0.015)	0.082/ 0.086 (0.084)	IB-2012-JLW-020; IB-2012-JLW-004-06 [Wiedmann and McDonald, 2014a]
Branchton, Ontario, Canada, 2013 (Ida Red)	3 (14)	102 102 101	7 7 7	BBCH 85-87 Sept 18	7	0.024/0.030 (0.027)	< 0.01/< 0.01(< 0.01)	0.035/ 0.041 (0.038)	IB-2013-JLW-004; IB-2013-JLW-004-05 [Wiedmann and McDonald, 2014a]

[ADJ] = adjuvant (NIS = non-ionic surfactant) added to the mix.

[a] mean of triplicate analyses

[b] single samples from two different plots at the same test site.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on apples in 17 European residue trials [Schäufele, 2013a, report JSM0347; Schäufele, 2013b, report JSM0348; Schäufele, 2013c, report JSM0473]. Two spray applications at 37–45 g ai/ha, with an interval of 13–15 days were applied at volumes corrected for tree size ranging from 474–1046 L/ha. Trials were performed in North and South Europe and include 8 decline trials. In one trial [Schäufele M, 2013b, JSM0348-04] a 3-fold exaggerated dose (127 and 124 g ai/ha) was applied for processing purposes. The results are summarised in Table 56.

Table 56 Supervised field trials on apples (fruit) treated with a pre-harvest foliar application with a formulation of cyclaniliprole (50SL)

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Niederrotweil, Germany, 2012 (Jonagold)	2 (15)	36.9 38.8	5 5	85 August, 29	0 2 7 9 13	0.04/0.03 (0.04) 0.02 0.01 0.03 0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.05/ 0.04 (0.05) 0.03 0.02 0.04 0.03/ 0.02 (0.03)	JSM0347; JSM0347-01; [Schäufele M, 2013a]
Impflingen, Germany, 2012 (Gala)	2 (13)	39 38.1	5 5	81 August 20	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01(< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0348; JSM0348-01; [Schäufele M, 2013b] see processing
Impflingen, Germany, 2012 (Elstar)	2 (14)	44.7 43.1	5 5	85 Sept 02	0 3 7 11 14	0.02/0.02 (0.02) 0.02 0.01 0.01 0.01/0.01 (0.01)	< 0.01/< 0.01(< 0.01) < 0.01 < 0.01 < 0.01/< 0.01(< 0.01)	0.03/ 0.03 (0.03) 0.03 0.02 0.02 0.02/ 0.02 (0.02)	JSM0473; JSM0473-01; [Schäufele M, 2013c]
Brumath, France (N) 2012 (Jonagored)	2 (14)	41.9 43.2	5 5	81-85 August 28	14	0.02/0.02 (0.02)	< 0.01/< 0.01(< 0.01)	0.03/ 0.03 (0.03)	JSM0348; JSM0348-02; [Schäufele M, 2013b] see processing
Loromontzey, France (N), 2012 (Braeburn)	2 (14)	39.8 41.3	5 5	85 Sept 19	0 2 7 9 13	0.04/0.04 (0.04) 0.03 0.01 < 0.01 0.01/0.01 (0.01)	< 0.01/< 0.01(< 0.01) < 0.01 < 0.01 < 0.01/< 0.01(< 0.01)	0.05/ 0.05 (0.05) 0.04 0.02 < 0.01 0.02/ 0.02 (0.02)	JSM0347; JSM0347-02; [Schäufele M, 2013a]
Rottelsheim, France (N), 2012 (Delbard estival)	2 (14)	39.3 38.6	8 8	81 July 26	14	0.02/0.01 (0.02)	< 0.01/< 0.01(< 0.01)	0.03/ 0.02 (0.03)	JSM0473; JSM0473-02; [Schäufele M, 2013c]
Skenfrith, UK, 2012 (Hastings)	2 (13)	43.3 38.2	5 5	83-85 Sept 06	0 5 7 12 14	0.06/0.04 (0.05) 0.02 [SS] 0.02 0.03 0.03/0.02 (0.03)	< 0.01/< 0.01 (< 0.01) < 0.01 [SS] < 0.01 < 0.01 0.01/< 0.01 0.01	0.07/ 0.05 (0.06) 0.03 0.03 0.04 0.041/ 0.03 (0.041)	JSM0347; JSM0347-03; [Schäufele M, 2013a]
Groesbeek, Netherlands, 2012 (Jonagold)	2 (13)	41.0 40.4	5 5	85 Sept 03	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01(< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0347; JSM0347-04; [Schäufele M, 2013a]
Grenade sur Garonne France (S), 2012 (Golden)	2 (15)	44.4 42.9	5 5	85 August 24	0 3 7 10 14	0.04/0.06 (0.05) 0.03 0.02 0.03 0.02/0.02 (0.02)	< 0.01/< 0.01(< 0.01) < 0.01 < 0.01 < 0.01/< 0.01(< 0.01)	0.05/ 0.07 (0.06) 0.04 0.03 0.04 0.03/ 0.03 (0.03)	JSM0347; JSM0347-05; [Schäufele M, 2013a]
Grenade sur Garonne, France (S), 2012	2 (14)	40.4 40.2	5 5	89 August 21	13	0.01/0.02 (0.02)[a]	< 0.01/< 0.01 (< 0.01)[a]	0.02/ 0.03 (0.03)[a]	JSM0348; JSM0348-03; [Schäufele M, 2013b]

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
(Golden)									see processing
Vacquiers, France (S), 2012 (Gala)	2 (15)	43.4 41.7	5 5	85 August 24	14	0.01/0.01 (0.01)	< 0.01/< 0.01(< 0.01)	0.02/ 0.02 (0.02)	JSM0347; JSM0347-06; [Schäufele M, 2013a]
Monteton, France (S), 2013 (Fuji)	2 (14)	39.4 37.9	5 5	85 Sept 13	14	0.01/0.01 (0.01)	< 0.01/< 0.01(< 0.01)	0.02/ 0.02 (0.02)	JSM0473; JSM0473-03; [Schäufele M, 2013c]
Vacquiers, France (S), 2013 (Gala)	2 (15)	40.3 41.5	4 4	81 August 06	0 3 7 10 14	0.04/0.06 (0.05) 0.02 0.01 0.02 0.02/0.02 (0.02)	< 0.01/< 0.01(< 0.01) < 0.01 < 0.01 < 0.01/0.01 (0.01)	0.05/ 0.07 (0.06) 0.03 0.02 0.03 0.03/ 0.03 (0.03)	JSM0473; JSM0473-04; [Schäufele M, 2013c]
Volpedo, Italy, 2012 (Golden)	2 (14)	40.6 41.7	5 5	85 August 22	0 2 7 10 13	0.02/0.03 (0.03) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01(< 0.01) < 0.01 < 0.01 < 0.01/< 0.01(< 0.01)	0.03/ 0.04 (0.04) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	JSM0347; JSM0347-07; [Schäufele M, 2013a]
San Sebastiano Curone, Italy, 2012 (Galaxy)	2 (14)	42.1 41.6	5 5	85 August 22	13	0.01/0.02 (0.02)	< 0.01/< 0.01(< 0.01)	0.02/ 0.03 (0.03)	JSM0348; JSM0348-04; [Schäufele M, 2013b]
idem	2 (14)	126.8 124.5	15 15	idem	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01(< 0.01)	< 0.01/< 0.01 (< 0.01)	idem see processing
Gualta, Spain, 2012 (Granny Smith)	2 (14)	42.5 44.5	5 5	89 Sept 24	0 3 7 9 14	0.02/0.02 (0.02) 0.02 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01(< 0.01) < 0.01 < 0.01 < 0.01/< 0.01(< 0.01)	0.03/ 0.03 (0.03) 0.03 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	JSM0347; JSM0347-08; [Schäufele M, 2013a]
Gualta, Spain, 2013 (Gala)	2 (14)	42.6 38.9	4 4	76-78 July 31	14	0.01/0.01 (0.01)	< 0.01/< 0.01(< 0.01)	0.02/ 0.02 (0.02)	JSM0473; JSM0473-05; [Schäufele M, 2013c]

[a] Carried out in the same field, on the same variety and approximately the same dates as the JSM0347-05. Not to be used for MRL derivation.

A 50 g/L soluble concentrate (IKI-3106 50 SL aka IBE 4064) was applied in 9 Australian field residue trials [Farrell, 2013a, report ULP-1113; Farrell, 2013b, report ISK12433]. Two spray applications at 27–312 g ai/ha, with an interval of 13–15 days were applied at volumes ranging from 690–3945 L/ha. Trials were performed in 2012 and 2013 growing seasons and include three decline trials. The results are summarised in Table 57.

Table 57 Supervised field trials on apples (fruit) treated with a pre-harvest foliar application with a formulation of cyclaniliprole (50SL)

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Applethorpe, Queensland, Australia, 2012 (Red delicious)	2 (14)	97.5 84.1	3.9 3.9	BBCH 75 Jan 17	0 7 14 21 28 35	0.062 0.032 0.029 0.02 0.021 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.073 0.043 0.040 0.031 0.032 0.021	ULP-1113; ULP-1113-01 [Farrell P, 2013a]
idem	idem	153 127	7.9 7.9	idem	0 7 14 21 28 35	0.17 0.05 0.039 0.033 0.04 0.032	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.18 0.061 0.050 0.044 0.051 0.043	idem
Yering, Victoria, Australia, 2012 (Fuji)	2 (15)	135 153	3.9 3.9	BBCH 78-81 March 02	0 7 14 21 28 34	0.11 0.045 0.031 0.033 0.025 0.023	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.12 0.056 0.042 0.044 0.036 0.034	ULP-1113; ULP-1113-02 [Farrell P, 2013a]
idem	idem	283312	7.9 7.9	idem	0 7 14 21 28 34	0.19 0.12 0.071 0.043 0.051 0.046	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.20 0.13 0.082 0.054 0.062 0.057	idem
Coldstream, Victoria, Australia, 2013 (Pink Lady)	2 (15)	117 134	3.9 3.9	BBCH 78 March 04	29	0.05	< 0.01	0.061	ISK12433; ISK12433-01; [Farrell P, 2013b]
idem	idem	241 267	7.7 7.7	idem	29	0.082	< 0.01	0.093	idem
Spreyton, Tasmania, Australia, 2013 (Golden delicious)	2 (14)	26.9 29.8	3.9 3.9	BBCH 77 Feb 13	28	< 0.01	< 0.01	< 0.01	ISK12433; ISK12433-02; [Farrell P, 2013b]
idem	idem	64.4 67.4	7.7 7.7	idem	28	< 0.01	< 0.01	< 0.01	idem
Paracoombe, South Australia, 2013 (Pink Lady)	2 (14)	78.7 79.8	3.9 3.9	BBCH 78 Feb 13	29	0.038	< 0.01	0.049	ISK12433; ISK12433-03; [Farrell P, 2013b]
idem	idem	165 145	7.7 7.7	idem	29	0.11	< 0.01	0.12	idem
Karragullen, West Australia, Australia, 2013 (Granny Smith)	2 (14)	59.1 58.3	3.9 3.9	BBCH 74-81 Feb 06	0 7 14 21 28 35	0.10 0.057 0.071 0.051 0.036 0.028	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.11 0.068 0.082 0.062 0.047 0.039	ISK12433; ISK12433-04; [Farrell P, 2013b]
idem	idem	128 131	7.7 7.7	idem	0 7 14 21 28	0.22 0.24 0.17 0.15 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.23 0.25 0.18 0.16 0.13	idem

APPLES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
					35	0.11	< 0.01	0.12	

Pears

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in field residue trials on apples in the USA (8) and in Canada (2) [Wiedmann and McDonald, 2014a, report IB-2013-JLW-004-01-01]. Three spray applications at 59–134 g ai/ha, with an interval of 6–14 days were applied with spray volumes ranging from 928–1813 L/ha. Trials were performed in the growing season 2013 and include one decline trial. The results are summarised in Table 58.

Table 58 Supervised field trials on pears (fruit) treated with a pre-harvest foliar application with a formulation of cyclaniliprole (50SL)

PEARS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Oswego, NY, USA, 2013 (Bartlett)	3 (14)	99 102 102	8 9 9	BBCH 81 Aug 12	7	0.050/0.069 (0.060)	< 0.01/0.012 (0.011)	0.061/ 0.080 (0.070)	IB-2013-JLW-004; IB-2013-JLW-004-08 [Wiedmann and McDonald, 2014a]
Conklin, MI, USA, 2013 (Bartlett)	3 (14)	100 100 100	6 6 6	BBCH 81-83 Aug 27	7	0.062/0.075 (0.069)	0.013/0.011 (0.012)	0.076/ 0.087 (0.081)	IB-2013-JLW-004; IB-2013-JLW-004-09 [Wiedmann and McDonald, 2014a]
Blissfield, MI, USA, 2013 (not reported)	3 (14, 6)	107 59 59	8 8 8	BBCH ripening Sept 3	15	< 0.01/< 0.01(< 0.01)	< 0.01/< 0.01(< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2013-JLW-004; IB-2013-JLW-004-10 [Wiedmann and McDonald, 2014a]
Madera, CA, USA, 2013 (Hosui)	3 (7, 7)	101 99 100	7 7 7	BBCH 77 Sept 05	7	0.082/0.11 (0.095)	0.012/0.011 (0.012)	0.095/0.12 (0.11)	IB-2013-JLW-004; IB-2013-JLW-004-13 [Wiedmann and McDonald, 2014a]
Lindsay CA, USA, 2013 (Olympic)	3 (14, 13)	99 100 100	4 4 4	BBCH 89 Sept 10	6	0.10/0.11 (0.11)	< 0.01/< 0.01(< 0.01)	0.11/ 0.12 (0.12)	IB-2013-JLW-004; IB-2013-JLW-004-14 [Wiedmann and McDonald, 2014a]
Ephrata, WA, USA, 2013 (D'Anjou)	3 (14)	98 99 99	11 11 11	BBCH 87-87 Aug 29	7	0.14/0.14 (0.14)	0.022/0.024(0.023)	0.16/ 0.16 (0.16)	IB-2013-JLW-004; IB-2013-JLW-004-15 [Wiedmann and McDonald,

PEARS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
									2014a]
Fruitland, ID, USA, 2013 (Bartlett)	3 (14, 13)	100 103 134	9 9 9	BBCH advanced fruit ripening Aug 21	7	0.094/0.100(0.097)	0.017/0.019(0.018)	0.11/ 0.12 (0.12)	IB-2012-JLW-020; IB-2012-JLW-004-16 [Wiedmann and McDonald, 2014a]
Hood River, OR, USA, 2013 (Star Crimson) + [ADJ]	3 (14)	100 101 102	7 7 7	BBCH 85 Sept 15	1 4 7 10	0.048/0.057(0.053) 0.041/0.042(0.042) 0.037/0.036 (0.037) 0.038/0.033(0.036)	0.010/0.015(0.013) 0.015/0.017 (0.016) 0.014/0.014 (0.014) 0.016/0.013 (0.015)	0.059/ 0.073 (0.066) 0.057/ 0.075 (0.066) 0.052/ 0.051 (0.051) 0.055/ 0.047 (0.051)	IB-2012-JLW-020; IB-2012-JLW-004-16 [Wiedmann and McDonald, 2014a]
Branchton, Ontario, Canada, 2013 (Bosc)	3 (14)	99 100 100	7 7 7	BBCH 85 Sept 18	7	0.12/0.13 (0.13)	0.016/0.016 (0.016)	0.14/ 0.15 (0.14)	IB-2013-JLW-004; IB-2013-JLW-004-11 [Wiedmann and McDonald, 2014a]
Arkona, Ontario, Canada, 2013 (Bosc)	3 (14)	99 101 101	7 7 7	BBCH fruit at full size, beginning to colour	7	0.13/0.15 (0.14)	0.017/0.019 (0.018)	0.15/ 0.17 (0.16)	IB-2013-JLW-004; IB-2013-JLW-004-12 [Wiedmann and McDonald, 2014a]

[ADJ] = adjuvant (NIS = non-ionic surfactant) added to the mix.

Stone fruit

Cherries

Supervised residue trials with applications of cylcaniliprole on cherries were performed in the USA, Canada and Japan.

A 50 g/L soluble concentrate (IKI-3106 50 SL aka IBE 4064) was applied on cherries (sweet cherries and tart cherries) in field residue trials in USA (15) and Canada (2) [Wiedmann & McDonald, report IB-2013-JLW-005]. Three spray applications of 91–117 g ai/ha with an interval of 6–8 days were applied at in a volume of 737–1526 L/ha. Trials were performed in 2013 growing season and include one decline trial. For analyses the fruits were de-pitted. Sample weights of whole fruit and de-pitted fruit were not reported. No conversion factor from flesh to RAC could be calculated. Results are only presented in flesh without stone. The results are summarised in Table 59.

Table 59 Supervised field trials on cherries (flesh (no stone)) treated with a pre-harvest foliar application with a formulation of cyclaniliprole (50SL)

CHERRIES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Conklin, MI, USA,	3 (7)	99 99	10 10	BBCH 85-87	7	0.37/0.23 (0.30)	0.017/0.011 (0.014)	0.39/ 0.24 (0.32)	IB-2013-JLW-005;

CHERRIES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2013 (Sam) + [ADJ] sweet cherry		99	10	July 01					IB-2013-JLW-005-22 [Wiedmann & McDonald, 2013c]
Blissfield, MI, USA, 2013 (variety not reported) sweet cherry	3 (6)	97 97 97	8 8 8	Ripening July 29	6	< 0.01/0.010 (0.010)	< 0.01/< 0.01 (< 0.01)	0.020/ 0.021 (0.021)	IB-2013-JLW-005; IB-2013-JLW-005-23 [Wiedmann & McDonald, 2013c]
Perry, UT, USA, 2013 (Bing) sweet cherry	3 (6)	105 103 102	7 7 7	BBCH 85 June 22	7	0.10/0.15 (0.13)	< 0.01/0.013 (0.012)	0.11/ 0.17 (0.14)	IB-2013-JLW-005; IB-2013-JLW-005-24 [Wiedmann & McDonald, 2013c]
Plainview, CA, USA, 2013 (Tulare) + [ADJ] sweet cherry	3 (7)	100 102 107	11 11 11	BBCH 87 May 08	7	0.36/0.30 (0.33)	< 0.01/< 0.01 (< 0.01)	0.37/ 0.31 (0.34)	IB-2013-JLW-005; IB-2013-JLW-005-25 [Wiedmann & McDonald, 2013c]
Fresno, CA, USA, 2013 (Brooks) + [ADJ] sweet cherry	3 (7)	98 99 98	13 13 13	Mature coloured fruit, at com-mercial harvest May 03	6	0.16/0.20 (0.18)	0.011/0.016 (0.014)	0.17/ 0.21 (0.19)	IB-2013-JLW-005; IB-2013-JLW-005-26 [Wiedmann & McDonald, 2013c]
Caldwell, ID, USA, 2013 (Bing) sweet cherry	3 (8)	103 104 100	8 8 8	BBCH 85 June 22	7	0.11/0.086 (0.097)	0.011/0.010 (0.011)	0.12/ 0.098 (0.11)	IB-2013-JLW-005; IB-2013-JLW-005-27 [Wiedmann & McDonald, 2013c]
Weisser, ID, USA, 2013 (Benton) + [ADJ] sweet cherry	3 (6-8)	103 105 102	7 7 7	Fruit colour advanced June 26	7	0.12/0.14 (0.13)	< 0.01/< 0.01 (< 0.01)	0.13/ 0.15 (0.14)	IB-2013-JLW-005; IB-2013-JLW-005-28 [Wiedmann & McDonald, 2013c]
Ephrata, WA USA,	3 (7)	104 99 99	11 11 11	BBCH 81 June 13	7	0.15/0.13 (0.14)	0.018/0.012 (0.015)	0.17/ 0.14 (0.16)	IB-2013-JLW-005; IB-2013-JLW-

CHERRIES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2013 (Bing) +[ADJ] sweet cherry									005-29 [Wiedmann & McDonald, 2013c]
Hereford, PA USA, 2013 (Mont-morency) tart cherry	3 (6)	103 102 101	12 12 12	BBCH 87 June 29	6	0.25/0.23 (0.24)	0.022/0.021 (0.022)	0.27/ 0.26 (0.26)	IB-2013-JLW-005; IB-2013-JLW-005-30 [Wiedmann & McDonald, 2013c]
Conklin, MI, USA, 2013 (Mont-morency) + [ADJ] tart cherry	3 (7)	99 99 99	10 10 10	BBCH 85-87 July 01	7	0.43/0.45 (0.44)	0.041/0.036 (0.039)	0.47/ 0.49 (0.48)	IB-2013-JLW-005; IB-2013-JLW-005-31 [Wiedmann & McDonald, 2013c]
Blissfield, MI, USA, 2013 (variety not reported) tart cherry	3 (7)	116 117 116	8 8 8	Ripening Aug 05	7	0.014/0.018 (0.016)	< 0.01/< 0.01 (< 0.01)	0.025/ 0.029 (0.027)	IB-2013-JLW-005; IB-2013-JLW-005-32 [Wiedmann & McDonald, 2013c]
Marengo, IL, USA, 2013 (North Star) tart cherry	3 (7)	99 100 99	8 8 8	Ripe July 02	7	0.081/0.082 (0.082)	0.020/0.021 (0.021)	0.10/ 010 (0.10)	IB-2013-JLW-005; IB-2013-JLW-005-34 [Wiedmann & McDonald, 2013c]
Perry, UT, USA, 2013 (Mont-morency) tart cherry	3 (6-7)	102 102 103	8 8 8	BBCH 87 July 09	6	0.28/0.29 (0.28)	0.050/0.054 (0.052)	0.33/ 0.35 (0.34)	IB-2013-JLW-005; IB-2013-JLW-005-35 [Wiedmann & McDonald, 2013c]
Branchton, Ontario, Canada, 2013 (North Star) + [ADJ] tart cherry	3 (7-8)	97 99 95	10 10 10	BBCH 78-79 June 18	4 7 10 14	0.34/0.28 (0.31) 0.14/0.14 (0.14) 0.090/0.081 (0.086) 0.071/0.074 (0.073)	0.064/0.064 (0.064) 0.030/0.031 (0.031) 0.025/0.020 (0.023) 0.017/0.019 (0.018)	0.40/ 0.35 (0.37) 0.17/ 0.17 (0.17) 0.12/ 0.10 (0.11) 0.089/ 0.094 (0.092)	IB-2013-JLW-005; IB-2013-JLW-005-33 [Wiedmann & McDonald, 2013c]
Josphberg, Alberta, Canada, 2013 (Evans)	3 (6-7)	91 110 100	10 11 10	BBCH 87-89 Aug 13	7	0.57/0.56 (0.56)	0.052/0.045 (0.049)	0.62/ 0.60 (0.61)	IB-2013-JLW-005; IB-2013-JLW-005-36 [Wiedmann &

CHERRIES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
tart cherry									McDonald, 2013c]

ADJ = Adjuvant (NIS = non-ionic surfactant) was added with application.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on cherries in field residue trials in Japan (2) [Kuzaki I and Naruto T, 2013]. Two spray applications of 104–114 g ai/ha with an interval of 7 days were applied at in a volume 4170–4550 L/ha (417–455 L/10 are). Trials were performed in the 2012 growing season and include two decline trials. The residues were measured in flesh only. The results are summarised in Table 60. According to the trial site information the trees were cultivated under plastic (from May onwards, suggesting a temporary coverage) in Fukushima and or continuously growing in a rain-protected house (Suzaka). It is not clear whether the trial represent indoor or outdoor trials.

Table 60 Supervised trials on cherries (fruit without stone) treated with a formulation with cyclaniliprole (50SL) using a pre-harvest foliar (backpack) sprayer

CHERRIES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Aza-Michima, Izakamachihirano, Fukushima-shia, Japan, 2012 (Sato Nishiki)	2 (7)	111 111	3 3	mature stage June 20	1 3 7	0.13/0.12 (0.12) 0.12/0.11 (0.12) 0.16/0.15 (0.16)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) 0.01/0.01 (0.01)	0.14/ 0.13 (0.13) 0.13/ 0.12 (0.12) 0.17/ 0.16 (0.17)	no report no.; Fukushima [Kuzaki I and Naruto T, 2013]
idem	idem	idem	idem	idem	1 3 7	0.15/0.13 (0.14) 0.13/0.12 (0.13) 0.18/0.17 (0.17)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) 0.011/0.011 (0.011)	0.16/ 0.15 (0.15) 0.15/ 0.14 (0.14) 0.19/ 0.18 (0.19)	corrected to whole fruit
idem	idem	111 111	3 3	young fruit period June 6	14 21	0.11/0.10 (0.11) 0.09/0.09 (0.09)	0.01/0.01 (0.01) 0.01/0.01 (0.01)	0.12/ 0.11 (0.12) 0.10/ 0.10 (0.10)	idem
idem	idem	idem	idem	idem	14 21	0.12/0.11 (0.12) 0.10/0.10 (0.10)	0.011/0.011 (0.011) 0.011/0.011 (0.011)	0.13/ 0.12 (0.13) 0.11/0.11 (0.11)	corrected to whole fruit
Ohshima, Kamitakai-gun-Obuse-machi, Nagano, 2012 (Seiko-Nishiki)	2 (7)	114 114	3 3	immediately before mature stage June 12	1 3 7	0.36/0.35 (0.36) 0.32/0.32 (0.32) 0.24/0.24 (0.24)	0.02/0.02 (0.02) 0.02/0.02 (0.02) 0.02/0.02 (0.02)	0.38/ 0.37 (0.38) 0.34/ 0.34 (0.34) 0.26/ 0.26 (0.26)	no report no.; Suzaka [Kuzaki I and Naruto T, 2013]
idem	idem	idem	idem	idem	1 3 7	0.41/0.40 (0.40) 0.37/0.37 (0.37) 0.27/0.27 (0.27)	0.023/0.023 (0.023) 0.023/0.023 (0.023) 0.023/0.023 (0.023)	0.43/ 0.43 (0.43) 0.39/ 0.39 (0.39) 0.30/ 0.30 (0.30)	corrected to whole fruit
idem		104	2	fruit	14	0.16/0.16	0.02/0.02	0.18/ 0.18	idem

CHERRIES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
		104	2	enlargement stage May 29	21	(0.16) 0.11/0.11 (0.11)	(0.02) 0.01/0.01 (0.01)	(0.18) 0.12/ 0.12 (0.12)	
idem	idem	idem	idem	idem	14 21	0.19/0.19 (0.19) 0.12/0.12 (0.12)	0.024/0.024 (0.024) 0.011/0.011 (0.011)	0.22/ 0.22 (0.22) 0.14/ 0.14 (0.14)	corrected to whole fruit

Plums

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in 16 European residue trials on plums [Schäufele, 2013i, report JSM0338; Schäufele, 2013j, report JSM0476]. Two spray applications with an interval of 13–15 days were applied at in a volume of 603–1594 L/ha. Trials were performed in North and South Europe and include 8 decline trials. Sample sizes consisted of at least 24 fruits and weighed above 2 kg unless indicated differently [SS]. Residues in whole fruit were calculated using the flesh/fruit ratio as determined by weighing. Ratio's ranged between 0.86 and 0.96. The results are summarised in Table 61.

Table 61 Supervised field trials on plums (whole fruit (calculated values based on ratio flesh/fruit) and flesh (no stone)) treated with two pre-harvest foliar applications of a formulation with cyclaniliprole (50SL)

PLUMS Location, Country; year; (variety)	No, (interval)	kg ai/ha	kg ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Niederrotweil, Germany, 2012 (Jojo)	2 (14)	41.1 40.2	4 4	BBCH 85 August 02	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0338; JSM0338-01; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem (flesh only)
Wendhausen, Germany, 2012 (Hauszwetschge)	2 (14)	37.3 37.8	3 3	BBCH 85 August 28	0 3 7 10 14	0.02/0.02 (0.02) [SS] 0.02 [SS] 0.01 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03) 0.03 0.02 0.02 < 0.01/< 0.01 (< 0.01)	JSM0338; JSM0338-02; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 10 14	na [SS] na [SS] 0.01 0.01 < 0.01/< 0.01 (< 0.01)	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.02 0.02 < 0.01/< 0.01 (< 0.01)	idem (flesh only)
Kerken, Germany, 2012 (Hauswetschge)	2 (15)	41.0 42.5	7 7	BBCH 85 August 29	0 3 7 10 14	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	JSM0338; JSM0338-05; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 10 14	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01	- - < 0.01 < 0.01 < 0.01/< 0.01	idem (flesh only)

PLUMS Location, Country; year; (variety)	No, (interval)	kg ai/ha	kg ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
						(< 0.01)	(< 0.01)	(< 0.01)	
Breisach, Germany, 2013 (Elena)	2 (14)	37.4 40.8	4 4	BBCH 81 August 13	0 3 7 10 14	0.04/0.04 (0.04) 0.03 0.01 0.02 0.01/< 0.01 (0.01)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.05) 0.04 0.02 0.03 0.02/< 0.01 (0.02)	JSM0476; JSM0476-02; [Schäufele M, 2013e] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 10 14	na na 0.01 0.02 0.01/< 0.01 (0.01)	na na < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.02 0.03 0.02/< 0.01 (0.02)	idem (flesh only)
Winchcombe, UK, 2012 (Victoria)	2 (13)	39.4 43.1	3 3	82 August 22	14	< 0.01/0.01 (0.01)[SS]	< 0.01 [SS]	< 0.01/0.02 (0.02)	JSM0338; JSM0338-06; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	14	< 0.01/0.01 [SS]	< 0.01/< 0.01 (< 0.01) [SS]	< 0.01/0.02 (0.02)	idem (flesh only)
Loromontzey, France (N), 2012 (Ortenauer)	2 (14)	41.4 40.6	4 4	BBCH 81 August 10	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0338; JSM0338-03; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem (flesh only)
Anthelupt, France (N), 2012 (Cacaks)	2 (14)	40.2 40.8	4 4	BBCH 81 August 10	0 3 7 10 13	0.05/0.04 (0.05) [SS] 0.02 [SS] 0.03 [SS] 0.02 < 0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 [SS] 0.01 < 0.01/< 0.01 (< 0.01)	0.06/ 0.05 (0.06) 0.03 0.04 0.03 < 0.01/0.02 (0.02)	JSM0338; JSM0338-04; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 10 13	na [SS] na [SS] 0.03 [SS] 0.02 < 0.01/< 0.01 (< 0.01)	na [SS] na [SS] < 0.01 [SS] < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.040 0.03 < 0.01/< 0.01 (< 0.01)	idem (flesh only)
Westhofen, France (N), 2013 (Ortenauer)	2 (14)	43.3 40.6	5 5	BBCH 81 August 13	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0476; JSM0476-01; [Schäufele M, 2013e] (whole fruit)
idem	idem	idem	idem	idem	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem (flesh only)
Aucamville, France (S), 2012 (Prune d'Ente)	2 (14)	39.7 39.7	4 4	BBCH 85 August 16	14	0.01/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.02/0.03 (0.03)	JSM0338; JSM0338-07; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	14	0.01/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.02/0.03 (0.03)	idem (flesh only)
Mas Grenier, France (S), 2012 (Prune d'Ente)	2 (14)	42.7 41.7	4 4	BBCH 85 August 16	0 3 7 10	0.03/0.03 (0.03) [SS] 0.01 [SS] 0.02 0.01	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01	0.04/ 0.04 (0.04) 0.02 0.03 0.02	JSM0338; JSM0338-08; [Schäufele M, 2013d] (whole fruit)

PLUMS Location, Country; year; (variety)	No, (interval)	kg ai/ha	kg ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
					14	0.01/< 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ < 0.01 (0.02)	
idem	idem	idem	idem	idem	0 3 7 10 14	na [SS] na [SS] 0.02 0.01 0.01/< 0.01 (0.01)	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.03 0.02 0.02/ < 0.01 (0.02)	idem (flesh only)
Aucamville, France (S), 2013 (Prune d'Ente)	2 (14)	40.1 40.4	4 4	BBCH 85 August 16	14	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	JSM0476; JSM0476-03; [Schäufele M, 2013e] (whole fruit)
idem	idem	idem	idem	idem	14	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	idem (flesh only)
Tassare di Avolasca, Italy, 2012 (President)	2 (13)	40.6 40.7	4 4	BBCH 87 August 16	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0338; JSM0338-09; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem (flesh only)
Brignano Frascata, Italy 2012 (Big Egg)	2 (14)	40.8 40.7	4 4	BBCH 81 July 25	0 3 7 9 14	0.03/0.03 (0.03) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04) < 0.01 < 0.01 < 0.01 < 0.01/ < 0.01 (< 0.01)	JSM0338; JSM0338-10; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 9 14	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	idem (flesh only)
Volpedo, Italy 2013 (Santa Clara)	2 (14)	41.0 43.3	3 3	BBCH 77 July 19	0 3 7 11 14	0.03/0.03 (0.03) 0.02 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) [SS]	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) [SS]	0.04/ 0.04 (0.04) 0.03 < 0.01 < 0.01 < 0.01/ < 0.01 (< 0.01)	JSM0476; JSM0476-04; [Schäufele M, 2013e] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 11 14	na na na na < 0.01/< 0.01 (< 0.01) [SS]	na na na na < 0.01/< 0.01 (< 0.01) [SS]	- - - - < 0.01/< 0.01 (< 0.01)	idem (flesh only)
Miravet - Tarragona, Spain 2012 (Angeleno)	2 (15)	38.7 40.9	4 4	BBCH 78-79 August 29	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0338; JSM0338-11; [Schäufele M, 2013d] (whole fruit)
idem	idem	idem	idem	idem	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem (flesh only)
Llambilles-Girona, 2012 (Golden globe)	2 (14)	41.2 39.8	4 4	BBCH 77-78 July 17	0 3 7 11 14	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 < 0.01 < 0.01/< 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01	JSM0338; JSM0338-12; [Schäufele M, 2013d] (whole fruit)

PLUMS Location, Country; year; (variety)	No, (interval)	kg ai/ha	kg ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
						< 0.01/< 0.01 (< 0.01)	(< 0.01)	(< 0.01)	
idem	idem	idem	idem	idem	0 3 7 11 14	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	na [SS] na [SS] < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	idem (flesh only)

[SS] samples weighed less than 2 kg, but at least 24 fruits were picked.

A 50 g/L soluble concentrate (IKI-3106 50 SL aka IBE 4064) was applied on plums in field residue trials in USA [Wiedmann & McDonald, report IB-2013-JLW-005]. Three spray applications of 40–103 g ai/ha with an interval of 6–8 days were applied at in a volume of 469–1503 L/ha. Trials were performed in 2013 growing season and include one decline trial. For analyses the fruits were de-pitted. Sample weights of whole fruit and de-pitted fruit were not reported. No conversion factor from flesh to RAC could be calculated. Results are only presented in flesh without stone. The results are summarised in Table 62. No weight fractions of pit to whole fruit and flesh were reported to calculate the residue level back to the RAC (inlcluding stone).

Table 62 Supervised field trials on plums (flesh, no stone) treated with a formulation with cyclaniliprole (50SL) using a pre-harvest foliar airblast sprayer

PLUMS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Blissfield, MI, USA, 2013 (not reported)	3 (7)	79 79 40	8 8 8	ripe August 28	6	0.027/0.027 (0.027)	0.013/0.014 (0.014)	0.041/ 0.042 (0.042)	IB-2013-JLW-005; IB-2013-JLW-005-14 [Wiedmann & McDonald, 2013c]
Conklin, MI, USA, 2013 (Stanley) + [ADJ]	3 (7)	100 99 99	10 10 10	BBCH 83-86 August 27	7	0.089/0.093 (0.091)	0.019/0.015 (0.017)	0.11/ 0.11 (0.11)	IB-2013-JLW-005; IB-2013-JLW-005-15 [Wiedmann & McDonald, 2013c]
Madera, CA, USA, 2013 (Apple Dandy) + [ADJ]	3 (7-8)	100 101 99	21 21 21	BBCH 79 May 23	6	0.023/0.024 (0.024)	< 0.01/< 0.01 (< 0.01)	0.034/ 0.035 (0.035)	IB-2013-JLW-005; IB-2013-JLW-005-16 [Wiedmann & McDonald, 2013c]
Orland, CA CA, USA, 2013 (French) + [ADJ]	3 (6-8)	99 100 100	11 11 11	50-60% purple August 05	7	0.018/0.019 (0.019)	< 0.01/< 0.01 (< 0.01)	0.029/ 0.030 (0.030)	IB-2013-JLW-005; IB-2013-JLW-005-17 [Wiedmann & McDonald, 2013c]
Lindsay, CA, USA,	3 (7)	100 100	10 10	BBCH 89	7	0.054/0.076 (0.065)	< 0.01/< 0.01 (< 0.01)	0.065/ 0.087	IB-2013-JLW-005;

PLUMS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2013 (Angelina's)		99	10	August 15				(0.076)	IB-2013-JLW-005-18 [Wiedmann & McDonald, 2013c]
Madera, CA, USA, 2013 (French – prune type) + [ADJ]	3 (7)	100 102 100	7 7 7	Mature prunes July 26	7	0.059/0.065 (0.062)	0.011/0.012 (0.012)	0.071/ 0.077 (0.075)	IB-2013-JLW-005; IB-2013-JLW-005-19 [Wiedmann & McDonald, 2013c]
idem	3 (7)	976 985 974	7 7 7	idem	7	0.81/0.67 (0.74)	0.16/0.11 (0.13)	0.97/ 0.79 (0.88)	idem, used for prune processing (see processing)
Payette, ID, USA, 2013 (Empress) + [ADJ]	3 (7)	103 102 101	11 11 11	advanced colouring of fruit August 26	0 1 7 10	0.066/0.089 (0.080) 0.089/0.064 (0.077) 0.049/0.063 (0.056) 0.040/0.046 (0.043)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.077/ 0.10 (0.091) 0.10/ 0.075 (0.088) 0.060/ 0.074 (0.067) 0.051/ 0.057 (0.054)	IB-2013-JLW-005; IB-2013-JLW-005-20 [Wiedmann & McDonald, 2013c]
Newberg, OR, USA, 2013 (Italian) + [ADJ]	3 (7)	99 99 100	7 7 7	BBCH 85-88 Sept 02	7	0.019/0.019 (0.019)	< 0.01/< 0.01 (< 0.01)	0.030/ 0.030 (0.030)	IB-2013-JLW-005; IB-2013-JLW-005-21 [Wiedmann & McDonald, 2013c]

[ADJ] = Adjuvant (NIS = non-ionic surfactant) was added with application.

Apricots, peaches and nectarines

No supervised residue trials were submitted for nectarines. The supervised residue trials with apricots (EU) and peaches (EU, USA and CAN) are summarised in the tables below.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue trials on apricots [Schäufele, 2013d, report JSM0329; Schäufele, 2013e, report JSM0474; Schäufele, 2014a, report JSM0667]. Two spray applications with an interval of 13–14 days were applied at in a volume of 907–1532 L/ha. Trials were performed in North and South Europe and include four decline trials. Sample sizes were above 2 kg unless indicated differently [SS]. Residues in whole fruit were calculated using the flesh/fruit weight ratio as determined by weighing. The flesh/fruit ratios for apricots in Europe ranged from 0.79–0.96 [studies JSM0329, JSM0474 and JSM0667]. The results are summarised in Table 63.

Table 63 Supervised field trials on apricots (whole fruit and flesh (no stone)) treated with a pre-harvest (foliar spray) formulation with cyclaniliprole (50SL)

APRICOTS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Kleve, Germany, 2014 (Goldrich)	2 (13)	41.1 40.3	3 3	BBCH 85 July 7	14	0.029/0.019 (0.024)	< 0.01/< 0.01 (< 0.01)	0.040/ 0.030 (0.035)	JSM0667; JSM0667-01; [Schäufele M, 2014h] (whole fruit)
idem	idem	idem	idem	idem	14	0.03/0.02 (0.03)	0.02/0.02 (0.02)	0.051/ 0.041 (0.051)	idem (flesh only)
Bedkowo, Poland, 2014 (Wczesna z Morden)	2 (14)	40.5 42.3	3 3	BBCH 76-77 June 18	0 8 11 14	0.04/0.03 (0.04) 0.02 [SS] < 0.01 [SS] < 0.01 < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01 [SS] < 0.01 [SS] < 0.01 < 0.01/< 0.01 (< 0.01)	0.05/ 0.04 (0.05) 0.03 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	JSM0667; JSM0667-02; [Schäufele M, 2014h] (whole fruit)
idem	idem	idem	idem	idem	0 2 8 11 14	na na < 0.01 [SS] < 0.01 [SS] < 0.01/< 0.01 (< 0.01)	na na < 0.01 [SS] < 0.01 [SS] < 0.01/< 0.01 (< 0.01)	- - < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	idem (flesh only)
Agárd, Hungary, 2013 (Gönci Magyarkajszi)	2 (13)	40.7 41.3	3 3	BBCH 74 June 21	0 3 7 10 14	0.08/0.10 (0.09) 0.05 0.03 [SS] 0.02 [SS] 0.02/0.02 (0.02) [SS]	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 [SS] < 0.01 [SS] < 0.01/< 0.01 (< 0.01) [SS]	0.09/ 0.11 (0.10) 0.06 0.04 0.03 0.03/ 0.03 (0.03)	JSM0474; JSM0474-01; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 10 14	na na 0.03 [SS] 0.02 [SS] 0.02/0.02 (0.02) [SS]	na na < 0.01 [SS] < 0.01 [SS] < 0.01/< 0.01 (< 0.01) [SS]	- - 0.04 0.03 0.03/ 0.03 (0.03)	idem (flesh only)
Veszprém, Hungary, 2013 (Magyarkajszi)	2 (13)	41.7 40.8	3 4	BBCH 78 June 19	14	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	JSM0474; JSM0474-02; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	14	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	idem (flesh only)
Montalzat, France (S), 2012 (Radirouge)	2 (14)	38.4 40.4	4 4	BBCH 81 July 24	0 3 7 10 14	0.06 0.05 0.03 0.06 0.04/0.02 (0.03)	< 0.01 < 0.01 < 0.01 0.01 0.02/< 0.01 (0.02)	0.07 0.06 0.04 0.071 0.061/ 0.03 (0.051)	JSM0329; JSM0329-01; [Schäufele M, 2013f] (whole fruit)
idem	idem	idem	idem	idem	0 3 7	na na 0.03	na na < 0.01	- - 0.04	idem (flesh only)

APRICOTS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
					10 14	0.06 0.04/0.02 (0.03)	0.01 0.02/< 0.01 (0.02)	0.071 0.061/ 0.03 (0.051)	
Pompignan, France (S), 2013 (Kioto)	2 (14)	435 42.6	4 4	BBCH 85 July 5	14	0.03/0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	JSM0474; JSM0474-03; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	14	0.03/0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	idem (flesh only)
Volpedo, Italy, 2012 (Bergero)	2 (14)	41.2 41.1	4 4	BBCH 83 June 21	15	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	JSM0329; JSM0329-02; [Schäufele M, 2013f] (whole fruit)
idem	idem	idem	idem	idem	15	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	idem (flesh only)
Garrigas, Spain, 2012 (Helena el Rossello)	2 (14)	36.9 41.4	4 4	BBCH 77-78 June 26	0 3 7 9 14	0.06/0.06 (0.06) 0.05 0.03 0.04 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.07/ 0.07 (0.07) 0.06 0.04 0.05 0.03/ 0.03 (0.03)	JSM0329; JSM0329-03; [Schäufele M, 2013f] (whole fruit)
idem	idem	idem	idem	idem	0 3 7 9 14	na na 0.03 0.04 0.02/0.02 (0.02)	na na < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.04 0.05 0.03/ 0.03 (0.03)	idem (flesh only)

na = not available.

[a] For some time points (0 and 14 days) duplicate field samples were taken. Results are presented as single values and (mean) of duplicate field samples.

[SS] samples size less than 2 kg, but at least 24 fruits were picked.

A 50 g/L soluble concentrate of cylcaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in eight European residue trials on peaches [Schäufele, 2013f, report JSM0351; Schäufele, 2013g, report JSM0352; Schäufele, 2013h, report JSM0475]. Two spray applications with an interval of 13–15 days were applied at in a volume of 961–1207 L/ha. Trials were performed in North and South Europe and include four decline trials. Residues in whole fruit were calculated using the flesh/fruit ratio as determined by weighing. Ratio's ranged between 0.85 and 0.96. The results are summarised in Table 64.

Table 64 Supervised field trials on peaches (whole fruit (calculated) and flesh (no stone)) treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

PEACHES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent [a]	NK-1375 [a]	Tot. [a]	
Tönisvorst, Germany, 2012 (Revit)	2 (13)	40.6 42.8	4 4	BBCH 85 July 18	0 2 6 9 13	0.02/0.04 (0.03) 0.04 0.02 < 0.01 0.01/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.03/ 0.05 (0.04) 0.05 0.03 < 0.01 0.02/ 0.03 (0.03)	JSM0352; JSM0352-01; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	0 2 6 9 13	na na 0.02 < 0.01 0.01/0.02 (0.02)	na na < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.03 < 0.01 0.02/ 0.03 (0.03)	idem (flesh only)
Neer, the Netherlands, 2012 (Revita)	2 (15)	41.2 39.6	4 4	BBCH 85 July 18	13	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	JSM0352; JSM0352-02; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	13	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02	idem (flesh only)
Loromontzey, France (N), 2013 (Sanguine Ferley)	2 (14)	39.1 42.6	4 4	BBCH 77 August 22	0 4 7 11 14	0.07/0.08 (0.08) 0.05 0.03 0.04 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.08/ 0.09 (0.09) 0.06 0.04 0.05 0.03/ 0.03 (0.03)	JSM0475; JSM0475-01; [Schäufele M, 2013h] (whole fruit)
idem	idem	idem	idem	idem	0 4 7 11 14	na na 0.03 0.04 0.02/0.02 (0.02)	na na < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.04 0.05 0.03/ 0.03 (0.03)	idem (flesh only)
Szekesfehervar, Hungary, 2013 (Cresthaven)	2 (14)	42.2 42.6	4 4	BBCH 79 July 31	15	0.03/0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	JSM0475; JSM0475-02; [Schäufele M, 2013h] (whole fruit)
idem	idem	idem	idem	idem	15	0.03/0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	idem (flesh only)
Gualta, Spain, 2013 (Rich Lady)	2 (14)	40.1 41.0	3 3	BBCH 76-77 June 21	0 4 7 10 14	0.10/0.08 (0.09) 0.04 0.04 0.02 0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.11/ 0.09 (0.09) 0.05 0.05 0.03 0.03/ 0.02 (0.03)	JSM0475; JSM0475-03; [Schäufele M, 2013h] (whole fruit)
idem	idem	idem	idem	idem	0 4 7 10 14	na na 0.04 0.02 0.02/0.01 (0.02)	na na < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.05 0.03 0.03/ 0.02 (0.03)	idem (flesh only)
Garrigas, Spain, 2012 (Summer Lady)	2 (14)	41.4 40.3	4 4	BBCH 75-76 July 19	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0351; JSM0351-02; [Schäufele M, 2013f] (whole fruit)

PEACHES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a] Parent [a]	NK-1375 [a]	Tot. [a]	Report; Trial no [ref]
idem	idem	idem	idem	idem	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem (flesh only)
Grenade sur Garonne, France (S), 2012 (Ronistar)	2 (14)	42.9 42.4	4 4	BBCH 81 July 6	13	0.02/< 0.01 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/< 0.01 (0.03)	JSM0352; JSM0352-03; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	13	0.02/< 0.01 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/< 0.01 (0.03)	idem (flesh only)
Volpedo, Italy, 2012 (Cresthaven)	2 (15)	40.7 39.9	4 4	BBCH 81 August 7	0 3 6 9 13	0.03/0.06 (0.05) 0.04 0.02 0.02 0.01/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.04/ 0.07 (0.06) 0.05 0.03 0.03 0.02/ 0.03 (0.03)	JSM0352; JSM0352-04; [Schäufele M, 2013g] (whole fruit)
idem	idem	idem	idem	idem	0 3 6 9 13	na na 0.02 0.02 0.01/0.02 (0.02)	na na < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	- - 0.03 0.03 0.02/0.03 (0.03)	idem (flesh only)
idem	idem	122 123	12 12	idem	13	0.08	0.03	0.11	idem (whole fruit) for processing, see processing section)

[a] For some time points (0 and 14 days) duplicate field samples were taken. Results are presented as single values and (mean) of duplicate field samples.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on peaches in field residue trials in USA and Canada [Wiedmann & McDonald, report IB-2013-JLW-005]. Three spray applications of 91–117 g ai/ha with an interval of 6–8 days were applied at in a volume of 737–1526 L/ha. Trials were performed in 2013 growing season and include one decline trial. For analyses the fruits were de-pitted. Sample weights of whole fruit and de-pitted fruit were not reported. No conversion factor from flesh to RAC could be calculated. Results are only presented in flesh without stone. The results are summarised in Table 65. No weight fractions of pit, whole fruit and/or flesh available for the purpose of calculating conversion factors to determine the residue level in the RAC including stone.

Table 65 Supervised field trials on peaches (flesh (no stone)) treated with a formulation with cyclaniliprole (50SL) using a pre-harvest foliar airblast sprayer

PEACHES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a] Parent [a]	NK-1375 [a]	Tot. [a]	Report; Trial no [ref]
Alton, NY, USA, 2013 (Baby Gold #5) + [ADJ]	3 (6-7)	100 100 101	11 11 11	BBCH 81 Aug 19	1 4 7 10	0.075/0.099 (0.087) 0.039/0.086 (0.063) 0.056/0.043 (0.050) 0.030/0.040 (0.035)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.086/ 0.11 (0.098) 0.050/ 0.097 (0.074) 0.067/ 0.054 (0.061) 0.041/ 0.051 (0.046)	IB-2013-JLW-005; IB-2013-JLW-005-01 [Wiedmann & McDonald, 2013c]

PEACHES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a]			Report; Trial no [ref]
						Parent [a]	NK-1375 [a]	Tot. [a]	
Plains, GA, USA, 2013 (Red Skin) + [ADJ]	3 (7)	102 102 103	8 8 11	BBCH 85 July 23	7	0.021/0.024 (0.023)	< 0.01/< 0.01 (< 0.01)	0.032/ 0.035 (0.034)	IB-2013-JLW-005; IB-2013-JLW-005-02 [Wiedmann & McDonald, 2013c]
Chula, GA, USA, 2013 (Gala) + [ADJ]	3 (7)	100 97 100	17 18 17	GS not reported June 5	7	0.083/0.078 (0.081)	< 0.01/< 0.01 (< 0.01)	0.094/ 0.089 (0.092)	IB-2013-JLW-005; IB-2013-JLW-005-03 [Wiedmann & McDonald, 2013c]
Monetta, SC, USA, 2013 (Big Red) + [ADJ]	3 (6-7)	99 97 97	9 9 9	BBCH 81 Aug 6	7	0.057/0.050 (0.054)	< 0.01/< 0.01 (< 0.01)	0.068/0.061 (0.065)	IB-2013-JLW-005; IB-2013-JLW-005-04 [Wiedmann & McDonald, 2013c]
Blissfield, MI, USA, 2013 (not reported)	3 (7)	79 79 55	8 8 8	fruits ripe Aug 28	7	0.014/0.023 (0.019)	< 0.01/< 0.01 (< 0.01)	0.025/ 0.034 (0.030)	IB-2013-JLW-005; IB-2013-JLW-005-06 [Wiedmann & McDonald, 2013c]
Conklin, MI, USA, 2013 (Red Haven) + [ADJ]	3 (7)	100 99 101	11 10 10	fruit tp 6.0 cm diameter Jul 31	6	0.16/0.22 (0.19)	< 0.01/0.012 (0.011)	0.17/ 0.23 (0.20)	IB-2013-JLW-005; IB-2013-JLW-005-07 [Wiedmann & McDonald, 2013c]
D'Hanis, TX, USA, 2013 (Flamin' Fury) + [ADJ]	3 (7)	99 99 100	11 11 8	BBCH 85 Jul 19	7	0.042 [SS]/0.039 (0.041)	0.017/0.015 (0.016)	0.060/ 0.055 (0.058)	IB-2013-JLW-005; IB-2013-JLW-005-09 [Wiedmann & McDonald, 2013c]
Madera, CA USA, 2013 (Spring Crest) + [ADJ]	3 (6-8)	98 101 99	13 13 13	Medium size fruit 70-80% ripe for harvest May 30	7	0.066/0.036 (0.051)	< 0.01/< 0.01 (< 0.01)	0.077/ 0.047 (0.062)	IB-2013-JLW-005; IB-2013-JLW-005-10 [Wiedmann & M7cDonald, 20173c]
Fresno, CA, USA, 2013 (Kaweah) + [ADJ]	3 (7)	100 99 99	11 11 11	BBCH 85 Jul 30	7	0.043/0.046 (0.045)	< 0.01/< 0.01 (< 0.01)	0.054/ 0.057 (0.056)	IB-2013-JLW-005; IB-2013-JLW-005-11 [Wiedmann & McDonald, 2013c]
Porterville, CA, USA, 2013 (Fay Alberta)	3 (7)	10010 0 99	11 11 11	BBCH 81 Jul 17	7	0.058/0.070 (0.064)	0.011/0.014 (0.013)	0.070/ 0.085 (0.078)	IB-2013-JLW-005; IB-2013-JLW-005-12 [Wiedmann & McDonald, 2013c]
Weiser, ID, USA, 2013	3 (7)	99 99 99	11 11 11	Advanced fruit colour Sep 02	6	0.16/0.15 (0.16)	< 0.01/< 0.01 (< 0.01)	0.17/ 0.16 (0.17)	IB-2013-JLW-005; IB-2013-JLW-005-13

PEACHES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a]			Report; Trial no [ref]
						Parent [a]	NK-1375 [a]	Tot. [a]	
(O'Henry) + [ADJ]									[Wiedmann & McDonald, 2013c]
Branchton, Ontario, Canada, 2013 (Reliance) + [ADJ]	3 (7)	101 104 103	10 10 10	BBCH 79-85 Aug 8	6	0.091/0.096 (0.094)	< 0.01/< 0.01 (< 0.01)	0.10/ 0.11 (0.10)	IB-2013-JLW-005; IB-2013-JLW-005-05 [Wiedmann & McDonald, 2013c]
Arkona area, Ontario, Canada, 2013 (Harmony)	3 (7)	101 100 101	22 22 22	fruit showing characteritic colour 90-100% of final size Aug 20	7	0.10/0.12 (0.11)	< 0.01/0.010 (0.010)	0.11/ 0.13 (0.12)	IB-2013-JLW-005; IB-2013-JLW-005-08 [Wiedmann & McDonald, 2013c]

[ADJ] = Adjuvant (NIS = non-ionic surfactant) was added with application.

[SS] = sample size was 1.8 kg and below the required 2 kg.

Berries and small fruits

Small fruit and vine climbing: grapes (table and wine grapes)

A 50 g/L soluble concentrate of cylcaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue trials on table grapes [Schäufele, 2013k, report JSM0330; Schäufele, 2013l, report JSM0477]. Two spray applications (35–39 g ai/ha) with an interval of 13–15 days were applied at in a volume of 693–869 L/ha. Trials were performed in South Europe and include four decline trials. The results are summarised in Table 66.

Table 66 Supervised field trials on table grapes (fruit) treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

TABLE GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Sammichele di Bari, Puglia, Italy, 2012 (Italia, white)	2 (13)	34.6 35.9	5 5	BBCH 89 October 11	0 1 3 6	0.28/0.20 (0.24) 0.23 0.20/0.17 (0.19) 0.18	< 0.01/< 0.01 (< 0.01) < 0.01 0.01/< 0.01 (< 0.01) < 0.01	0.29/ 0.21 (0.25) 0.24 0.21/ 0.18 (0.20) 0.19	JSM0330; JSM0330-01; [Schäufele M, 2013k]
Mottola, Puglia, Italy, 2012 (Italia, white)	2 (15)	36.6 35.5	5 5	BBCH 89 October 14	3	0.07/0.07 (0.07)	0.01/< 0.01 (< 0.01)	0.081/ 0.081 (0.081)	JSM0330; JSM0330-02; [Schäufele M, 2013k]
Mottola, Puglia, Italy, 2013 (Vittoria, white)	2 (13)	36.4 38.7	4 4	BBCH 89 August 12	0 1 3 8	0.12/0.10 (0.11) 0.08 0.10/0.10 (0.10) 0.06	0.01/< 0.01 (< 0.01) < 0.01 0.01/< 0.01 (0.01) < 0.01	0.13/ 0.11 (0.12) 0.091 0.11/0.11 (0.11) 0.071	JSM0477; JSM0477-01; [Schäufele M, 2013l]

TABLE GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Padules, Almeria, Spain, 2012 (Napoleon, red)	2 (14)	37.5 37.0	5 5	BBCH 88 Sept. 5	0 1 3 7	0.21/0.15 (0.18) 0.22 0.24/0.17 (0.21) 0.11	0.01/< 0.01 (< 0.01) 0.01 0.01/< 0.01 (0.01) 0.01	0.22/ 0.16 (0.19) 0.23 0.25/ 0.18 (0.22) 0.12	JSM0330; JSM0330-03; [Schäufele M, 2013k]
Huecija, Almeria, Spain, 2012 (Red Globes, red)	2 (14)	36.5 36.8	5 5	BBCH 88 Sept. 5	3	0.07/0.12 (0.10)	0.01/< 0.01 (< 0.01)	0.081/ 0.13 (0.11)	JSM0330; JSM0330-04; [Schäufele M, 2013k]
Dalias, Almeria, Spain, 2013 (superior, white)	2 (14)	36.2 35.3	5 5	BBCH 85 July 26	3	0.06/0.06 (0.06)	0.01/< 0.01 (0.01)	0.071/ 0.071 (0.071)	JSM0477; JSM0477-02; [Schäufele M, 2013l]
Nea Gonia, Macedonia, Greece, 2012 (Razaki, white)	2 (15)	36.1 34.7	5 5	BBCH 85 Sept. 4	0 1 3 8	0.06/0.06 (0.06) 0.06 0.05/0.04 (0.05) 0.03	0.01/< 0.01 (< 0.01) 0.01 0.01/< 0.01 (0.01) < 0.01	0.071/ 0.071 (0.071) 0.071 0.061/0.051 (0.061) 0.041	JSM0330; JSM0330-05; [Schäufele M, 2013k]
Agios, Macedonia, Greece 2012 (Soultania, white)	2 (14)	35.5 34.7	5 5	BBCH 89 August 17	3	0.01/< 0.01 (0.01)	0.01/< 0.01 (< 0.01)	0.021/< 0.01 (0.021)	JSM0330; JSM0330-06; [Schäufele M, 2013k]

[a] For some time points (0 and 3 DALT) duplicate field samples were taken. Results are presented as single values and (mean) of duplicate field samples.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue trials on wine grapes [Schäufele, 2013m, report JSM0349; Schäufele, 2013n, report JSM0350, Schäufele, 2013o, report JSM0478]. Two spray applications (32–39 g ai/ha) with an interval of 13–15 days were applied at in a volume of 378–1083 L/ha. Trials were performed in South Europe and include 8 decline trials and some included plots for processing. The results are summarised in Table 67.

Table 67 Supervised field trials on wine grapes (fruit) treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

WINE GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Hammelburg Germany, 2012 (Regent, red)	2 (15)	35.1 35.4	4 4	BBCH 83-85 August 28	0 6 14 21 28	0.09/0.08 (0.09) 0.08 0.06 0.03 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) 0.01 0.01 0.01 < 0.01/< 0.01 (< 0.01)	0.10/0.09 (0.10) 0.09 0.07 0.04 0.03/0.03 (0.03)	JSM0349; JSM0349-01; [Schäufele M, 2013m]
Schweigen, Germany 2012, (Riesling, white)	2 (14)	32.7 33.3	4 4	BBCH 83 August 30	0 6 14 20 28	0.11/0.10 (0.11) 0.12 0.07 0.05 0.05/0.05	< 0.01/< 0.01 (< 0.01) 0.01 0.02 < 0.01 < 0.01/< 0.01	0.12/ 0.11 (0.12) 0.13 0.09 0.06 0.06/ 0.06	JSM0349; JSM0349-02; [Schäufele M, 2013m]

WINE GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
						(0.05)	(< 0.01)	(0.06)	
Vogstburg – Oberbergen, Germany, 2012 (Burgunder, white)	2 (14)	36.4 32.3	7 7	BBCH 83 August 28	27	0.03/0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.05 (0.05)	JSM0350; JSM0350-01; [Schäufele M, 2013n]
Bickensohl, Germany, 2013 (Domfelder, red)	2 (14)	33.7 32.8	4 4	BBCH 83 August 23	27	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	JSM0478; JSM0478-02; [Schäufele M, 2013o]
Westhofen France (N), 2012 (Pinot blanc, white)	2 (15)	37.3 36.4	7 7	BBCH 79-81 August 16	0 6 13 20 27	0.07/0.08 (0.08) 0.03 0.03 0.02 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.08/ 0.09 (0.09) 0.04 0.04 0.03 0.03/ 0.03 (0.03)	JSM0349; JSM0349-03; [Schäufele M, 2013m]
Bruley, France (N), 2012 (Auxerrois, white)	2 (14)	36.7 39.1	7 7	BBCH 77 Sept. 5	29	0.06/0.06 (0.06)	0.01/< 0.01 (0.01)	0.07/ 0.07 (0.07)	JSM0350; JSM0350-02; [Schäufele M, 2013n]
Furdenheim, France (N), 2012 (Pinot noir, red)	2 (15)	36.5 35.0	7 7	BBCH 79-81 August 16	27	0.04/0.03 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/0.04 (0.05)	JSM0350; JSM0350-03; [Schäufele M, 2013n]
Furdenheim. France (N), 2013 (Auxerrois, white)	2 (15)	33.7 34.3	9 9	BBCH 81 August 22	0 6 14 21 28	0.14/0.09 (0.12) 0.05 0.06 0.05 0.04/0.04 (0.04)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 0.01 < 0.01/< 0.01 (< 0.01)	0.15/ 0.10 (0.13) 0.06 0.07 0.06 0.05/ 0.05 (0.05)	JSM0478; JSM0478-01; [Schäufele M, 2013o]
Fronton, France (S), 2012 (Pinot noir, red)	2 (13)	34.3 33.6	7 7	BBCH 83 August 13	0 7 14 21 28	0.08/0.08 (0.08) 0.08 0.06 0.03 0.02/0.03 (0.03)	< 0.01/< 0.01 (< 0.01) 0.01 < 0.01 < 0.01 < 0.01	0.09/0.09 (0.09) 0.09 0.07 0.04 0.03/ 0.04 (0.04)	JSM0349; JSM0349-04; [Schäufele M, 2013m]
Tecou, France (S), 2012 (Merlot, red)	2 (13)	36.2 35.6	7 7	BBCH 83 August 13	28	0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.02 (0.03)	JSM0350; JSM0350-04; [Schäufele M, 2013n]
Fronton. France (S), 2013 (Sauvignon blanc, white)	2 (14)	38.6 34.4	4 4	BBCH 83 August 13	28	0.04/0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.06 (0.06)	JSM0478; JSM0478-03; [Schäufele M, 2013o]
Stenimachos, Greece, 2012 (Merlot, red)	2 (14)	34.3 33.6	7 7	BBCH 83 August 13	0 7 14 21 28	0.06/0.07 (0.07) 0.04 0.02 0.02 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.07/ 0.08 (0.08) 0.05 0.03 0.03 0.03/0.03 (0.03)	JSM0349; JSM0349-06; [Schäufele M, 2013m]
Casaeso, Italy, 2012 (Timorasso, white)	2 (15)	35.9 36.5	4 4	BBCH 87 Sept. 8	23	0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.02 (0.03)	JSM0350; JSM0350-05; [Schäufele M, 2013n]
Mornico Losana,	2	36.7	7	BBCH 83	0	0.15/0.15	< 0.01/< 0.01	0.16/0.16	JSM0478;

WINE GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Italy, 2013 (Merlot, red)	(14)	38.5	7	August 9	7 14 21 28	(0.15) 0.09 0.06 0.05 0.04/0.04 (0.04)	(< 0.01) 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	(0.16) 0.10 0.07 0.06 0.05/ 0.05 (0.05)	JSM0478-04; [Schäufele M, 2013o]
Garriguella, Spain, 2012 (Carinyena, red)	2 (14)	34.6 34.5	4 4	BBCH 81 August 27	28	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0350; JSM0350-06; [Schäufele M, 2013n]
Mollet de Peralada, Spain 2013 (Macabeu, white)	2 (15)	36.4 37.7	4 4	BBCH 79-81 July 30	0 7 14 22 28	0.06/0.05 (0.06) 0.03 0.03 0.02 0.02/0.03 (0.03)	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01)	0.07/ 0.06 (0.07) 0.04 0.04 0.06 0.03/ 0.04 (0.04)	JSM0478; JSM0478-05; [Schäufele M, 2013o]

[a] For some time points (0 and 28 DALT) duplicate field samples were taken. Results are presented as single values and (mean) of duplicate field samples.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on grapes in field residue trials in USA and Canada [McDonald & Wiedmann, report IB-2013-JAM-002]. Three spray applications of 97–105 g ai/ha with an interval of 6–8 days were applied at in a volume of 472–902 L/ha. No adjuvant was added to the mix in any of the trials. Trials were performed in 2013 growing season and include one decline trial. The results are summarised in Table 68.

Table 68 Supervised field trials on grapes treated with a formulation with cyclaniliprole (50SL) using a pre-harvest foliar airblast sprayer

GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Dundee, NY, USA, 2013 (Concord)	3 (7)	100 101 100	11 11 11	BBCH 85 Sept 20	6	0.21/0.20 (0.20)	< 0.01/< 0.01 (< 0.01)	0.22/ 0.21 (0.22)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-01 [McDonald &Wiedmann, 2014a]
Breinigsville, PA, USA, 2013 (Corot Noir)	3 (6-7)	100 100 100	13 13 13	BBCH 85 Sept 18	3 5 7 9	0.16/0.25 (0.20) 0.12/0.11 (0.11) 0.090/0.13 (0.11) 0.11/0.093 (0.10)	0.020/0.031 (0.026) 0.015/0.012 (0.014) 0.014/0.017 (0.016) 0.019/0.013 (0.016)	0.18/ 0.29 (0.23) 0.13/ 0.12 (0.13) 0.10/ 0.15 (0.13) 0.13/ 0.11 (0.12)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-02 [McDonald &Wiedmann, 2014a]
Oregon, WI, USA, 2013 (Concord)	3 (6-8)	100 100 100	12 12 12	BBCH 87 Sept 24	7	0.12/0.11 (0.12)	< 0.01/< 0.01 (< 0.01)	0.13/ 0.12 (0.13)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-05 [McDonald &Wiedmann, 2014a]
Fresno, CA, USA, 2013 (Flame seedless)	3 (7)	97 99 100	13 13 13	BBCH 83 August 28	7	0.022/0.027 (0.025)	< 0.01/< 0.01 (< 0.01)	0.033/ 0.038 (0.036)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-06 [McDonald &Wiedmann, 2014a]

GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/ hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Orland, CA, USA, 2013 (Rubired)	3 (7)	100 100 100	14 14 14	BBCH 85 July 30	7	0.15/0.12 (0.14)	0.033/0.032 (0.033)	0.19/ 0.16 (0.17)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-07 [McDonald &Wiedmann, 2014a]
Arbuckle, CA, USA, 2013 (Zinfandel)	3 (7)	100 100 100	12 14 14	BBCH 85 August 2	7	0.051/ 0.037 (0.044)	< 0.01/< 0.01 (< 0.01)	0.062/ 0.048 (0.055)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-08 [McDonald &Wiedmann, 2014a]
Kingsburg, A, USA, 2013 (Muscat)	3 (7)	99 103 102	12 14 14	BBCH 85 Sept 5	7	0.054/ 0.042 (0.048)	< 0.01/< 0.01 (< 0.01)	0.065/ 0.053 (0.059)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-09 [McDonald &Wiedmann, 2014a]
Porterville, CE, USA, 2013 (Thompson)	3 (6-7)	102 99 99	15 14 15	BBCH 85 August 8	7	0.061/ 0.091 (0.076)	0.011/0.018 (0.015)	0.072/ 0.11 (0.092)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-10 [McDonald &Wiedmann, 2014a]
Madera, CA, USA, 2013 (Thompson seedless)	3 (7)	105 102 102	12 12 12	mature grapes August 15	7	0.38/0.27 (0.33)	0.15/0.070 (0.11)	0.53/ 0.35 (0.44)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-11 [McDonald &Wiedmann, 2014a]
Kerman, CA, USA, 2013 (Thompson seedless)	3 (6-7)	103 102 102	12 12 12	BBCH 85-89 August 9	7	0.13/0.11 (0.12)	0.033/0.023 (0.028)	0.17/ 0.13 (0.15)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-12 [McDonald &Wiedmann, 2014a]
Creston, CAS, USA, 2013 (Cabernet Sauvignon)	3 (7-8)	100 100 100	18 18 18	BBCH 89 August 28	7	0.12/0.21 (0.16)	0.081/0.14 (0.11)	0.20/ 0.35 (0.28)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-13 [McDonald &Wiedmann, 2014a]
Ephrata, WA, USA, 2013 (White Riesling)	3 (7)	100 99 101	13 13 13	BBCH 85 Sept 17	7	0.36/0.41 (0.39)	0.090/0.089 (0.090)	0.46/ 0.51 (0.48)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-14 [McDonald &Wiedmann, 2014a]
New Plymouth, ID, USA, (Alborz)	3 (6-7)	100 101 100	21 21 21	advanced colour of berries, August 21	7	0.13/0.14 (0.14)	< 0.01/0.020 (0.015)	0.14/ 0.16 (0.15)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-15 [McDonald &Wiedmann, 2014a]
Branchton, Ontario Canada, 2013 (Concord)	3 (6-7)	98 103 98	12 12 12	BBCH 85-89Sept 19	7	0.25/0.24 (0.24)	< 0.01/0.01 (< 0.01)	0.26/ 0.25 (0.25)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-03 [McDonald &Wiedmann, 2014a]

GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Sparta, Ontario, Canada, 2013 (Aurore)	3 (7)	100 99 100	12 12 12	Fruit turning colour, 70-95% of final size August 20	7	0.54/0.48 (0.51)	0.087/0.074 (0.081)	0.63/ 0.55 (0.59)	IB-2013-JAM-002-01-01; IB-2013-JAM-002-04 [McDonald &Wiedmann, 2014a]

A 50 g/L soluble concentrate of cyclaniliprole was applied on grapes in field residue trials in Japan [Kimikazu, 2013a, report JP2012C100a and Kimikazu, 2013b, report JP2013C280]. Two foliar spray applications of 76–88 g ai/ha with an interval of 7 days were applied at in a volume 3020–3500 L/ha (302–350 L/10 are) with a back spray. Trials were performed in the 2012 and 2013 growing seasons and include four decline trials; sampling days 1, 3, and 7 on plot B and day 14 on plot C. The results are summarised in Table 69.

Table 69 Supervised trials on grapes treated with a formulation with cyclaniliprole (50SL) using a pre-harvest foliar (backpack) sprayer

GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Takaki, Hanamaki-shi, Iwate, Japan, 2012 (Beni-Izu)	2 (7)	87 87	3 3	harvesting phase August 31	1 3 7	0.09/0.08 (0.09) 0.08/0.08 (0.08) 0.11/0.11 (0.11)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.10/ 0.09 (0.10) 0.09/ 0.09 (0.09) 0.12/ 0.12 (0.12)	JP2012C100; JP2012C100A [Kimikazu, 2013a]
idem	2 (7)	87 87	3 3	Fruit colouring phase August 24	14	0.09 / 0.07 (0.08)	0.01/0.01 (0.01)	0.10/ 0.08 (0.09)	JP2012C100; JP2012C100A [Kimikazu, 2013a]
Futatsuya, Kahoku-shi, Ishikawa, Japan, 2012 (Delaware)	2 (7)	83 83	2 2	Fruit maturing stage July 09	1 3 7	0.46/0.45 (0.46) 0.41/0.40 (0.41) 0.31/0.30 (0.31)	< 0.01/< 0.01 (< 0.01) 0.01/< 0.01 (0.01) 0.01/0.01 (0.01)	0.47/ 0.46 (0.47) 0.42/ 0.41 (0.42) 0.32/ 0.31 (0.32)	JP2012C100; JP2012C100B [Kimikazu, 2013a]
idem	2 (7)	83 83	2 2	fruit colouring phase July 02	14	0.29 / 0.29 (0.29)	0.01/0.01 (0.01)	0.30/ 0.30 (0.30)	JP2012C100; JP2012C100B [Kimikazu, 2013a]
596-3 Daiku, Ymanashi-shi, Yamanashi, Japan (Delaware)	2 (7)	76 76	3 3	initial harvesting stage July 24	1 3 7	0.42/0.41 (0.42) 0.50/0.48 (0.49) 0.37/0.35 (0.36)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) 0.01/0.01 (0.01)	0.43/0.42 (0.43) 0.51/ 0.49 (0.50) 0.38/0.36 (0.37)	JP2012C100; JP2012C100C [Kimikazu, 2013a]
idem	2 (7)	76 76	3 3	colouring phase July17	14	0.25/0.23 (0.24)	< 0.01/< 0.01 (< 0.01)	0.26/0.24 (0.25)	JP2012C100; JP2012C100C [Kimikazu, 2013a]
Takanashi, Ooaza, Suzaka-shi,	2 (7)	88 88	3 3	colouring phase period	1 3	0.26/0.25 (0.26) 0.26/0.25	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01	0.27/ 0.26 (0.27) 0.27/ 0.26	JP2013C280; JP2013C280A [Kimikazu,

GRAPES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Nagano, Japan, 2013 (Kyoho)				July 15	7	(0.26) 0.29/0.27 (0.28)	(0.01) < 0.01/< 0.01 (< 0.01)	(0.27) 0.3/0.28 (0.29)	2013b]
idem	2 (7)	88 88	3 3	colouring phase July 01	14 21	0.25/0.23 (0.24) 0.24/0.24 (0.24)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.26/ 0.24 (0.25) 0.25/ 0.25 (0.25)	JP2013C280; JP2013C280C [Kimikazu, 2013b]

Brassicas

Flowerhead Brassicas

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue trials on broccoli [Alé, 2013a, report JSM0333, Alé, 2013b, report JSM0481]. Two knapsack or boomspray applications (24–29 g ai/ha) with an interval of 13–14 days were applied at in a volume of 202–313 L/ha at BBCH 39–46, apart from one trial in Spain (JSM0481-03) where the last treatment was applied at BBCH 22. Trials were performed in North and South Europe in the 2012 and 2013 growing seasons and include four decline trials. The results are summarised in Table 70.

Table 70 Supervised field trials on broccoli treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

BROCCOLI Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Wunstorf, Germany 2012 (Ironman)	2 (14)	26.6 25.7	13 13	BBCH 41-45 Oct 2	0 3 7 14 20	0.09/0.09 (0.09) 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.10/0.10 (0.10) 0.02 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0333; JSM0333-01; [Alé E, 2013a]
Innenheim, France (North), 2012 (Ronman)	2 (14)	24.2 26.6	10 10	BBCH 41-45 Oct 1	0 3 7 15 21	0.07/0.11 (0.09) 0.03 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.08/ 0.12 (0.10) 0.04 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0333; JSM0333-02; [Alé E, 2013a]
Boston, United Kingdom, 2012 (Steel)	2 (14)	25.5 25.5	10 10	BBCH 43-45 Oct 29	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0333; JSM0333-03; [Alé E, 2013a]
Wunstorf, Germany, 2013 (Ironman)	2 (14)	26.7 27.4	10 10	BBCH 39-41 Aug 09	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0481; JSM0481-01; [Alé E, 2013b]
Piemonte, Italy, 2012 (Green commet)	2 (13)	29.2 24.9	10 10	BBCH 46 Oct 18	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0333; JSM0333-05; [Alé E, 2013a]
Canjayar,	2	27.1	10	BBCH	0	0.03/0.03	< 0.01/< 0.01	0.04/0.04	JSM0333;

BROCCOLI Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Spain, 2012 (Nubia)	(13)	25.0	10	44 Nov 23	3 8 15 22	(0.03) 0.03 0.01 0.01/< 0.01 (0.01) < 0.01	(< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	(0.04) 0.04 0.02 0.02/< 0.01 (0.02) < 0.01	JSM0333-06; [Alé E, 2013a]
Tzermiadon, Greece, 2013 (Martor F1)	2 (14)	26.6 25.8	8 8	BBCH 44 Sept 16	0 4 7 15 21	0.04/0.05 (0.05) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.05/ 0.06 (0.06) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0481; JSM0481-02; [Alé E, 2013b]
Torroella de Montgo, Spain, 2013 (Trevy)	2 (14)	24.0 26.5	10 10	BBCH 22 Mar 13	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0481; JSM0481-03; [Alé E, 2013b]

[ADJ] = Adjuvant (R-11 Adjuvant (site 18 and 19)) was added with application.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on broccoli in field residue trials in USA and Canada [Wiedmann & McDonald, 2014b, report IB-2013-JLW-028-01-01]. Three spray applications of 61–87 g ai/ha with an interval of 6–8 days were applied at in a volume of 184–299 L/ha. An adjuvant was added to the mix at two test sites. Trials were performed in 2012 and 2013 growing seasons and include one decline trial. Sample sizes included 12 plants, weighing 0.49–5.7 kg. The results are summarised in Table 71.

Table 71 Supervised field trials on broccoli treated with a formulation with cyclaniliprole (50SL) using a pre-harvest back pack or tractor mounted side boom sprayer

BROCCOLI Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Seymour, IL, USA, 2013 (Waltham 29)	3 (7)	79 78 77	28 28 29	BBCH 66 Aug 22	1	0.31/0.52 (0.42)	0.059/0.086 (0.073)	0.37/ 0.61 (0.49)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-13 [Wiedmann &McDonald2014b]
York, NE, USA, 2013 (Coronade Crown hybrid)	3 (6-7)	79 79 78	38 38 38	BBCH 48 Oct 01	1	0.26/0.55 (0.41)	< 0.01/0.014 (0.012)	0.28/ 0.57 (0.42)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-14 [Wiedmann &McDonald2014b]
Hinton, OK, USA, 2013 (Calabrese)	3 (6-8)	82 80 77	39 41 42	BBCH 49-55 May 30	1	0.40/0.54 (0.47)	0.054/0.063 (0.059)	0.46/ 0.61 (0.54)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-15 [Wiedmann &McDonald2014b]
Porterville, CA, USA,	3 (7)	82 82	27 28	BBCH 49 May 09	1	0.13/0.088 (0.11)	0.010/< 0.01 (0.010)	0.14/ 0.099 (0.12)	IB-2012-JLW-028-01-01

BROCCOLI Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2013 (Heritage)		81	27						IB-2012-JLW-028-01-01-16 [Wiedmann &McDonald2014b]
Sanger, CA, USA, 2013 (Marathon)	3 (7)	80 87 83	29 29 29	BBCH 49 Feb 21	1	0.50/0.82 (0.66)	0.042/ 0.057 (0.050)	0.54/ 0.88 (0.71)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-17 [Wiedmann &McDonald2014b]
Blythe, CA, USA, 2013 (Green Goliath) + [ADJ]	3 (7)	61 61 61	32 33 33	BBCH 89 Jan 10	1	0.14/0.21 (0.18)	< 0.01/< 0.01(< 0.01)	0.15/ 0.22 (0.19)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-18 [Wiedmann &McDonald2014b]
Madera, CA, USA, 2013 (Heritage) + [ADJ]	3 (7)	63 61 61	22 22 22	Mature Broccoli heads 8-15 cm diameter Dec 25	1	0.11/0.12 (0.12)	< 0.01/< 0.01(< 0.01)	0.12/ 0.13 (0.13)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-19 [Wiedmann &McDonald2014b]
Hillsboro, OR, USA, 2013 (Bay Meadows)	3 (7)	81 80 80	34 34 34	BBCH 87 Aug 02	1	0.38/0.37 (0.37)	0.010/0.011 (0.011)	0.39/ 0.38 (0.38)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-20 [Wiedmann &McDonald2014b]
Branchton, Ontario, CANADA, 2013 (Imperial)	3 (7-8)	83 79 79	40 40 40	BBCH 49-63 Sept. 04	1 3 5 7	0.33/0.36 (0.34) 0.096/0.097 (0.097) 0.069/0.078 (0.074) 0.062/0.052 (0.057)	0.035 /0.043 (0.039) 0.015/0.016 (0.016) 0.011/0.012 (0.012) 0.011/< 0.01 (0.011)	0.36/ 0.40 (0.38) 0.11/ 0.11 (0.11) 0.081/0.091 (0.087) 0.073/ 0.063 (0.068)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-11 [Wiedmann &McDonald2014b]
St-Marc-sur-Richelieu, QC CANADA 2013 (Imperial)	3 (7)	79 81 82	40 40 40	BBCH 47-49 July 25	1	0.20 / 0.20 (0.20)	0.031 /0.029 (0.030)	0.23/ 0.23 (0.23)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-12 [Wiedmann &McDonald2014b]

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in eight European residue trials on cauliflower [Alé, 2013e, report JSM0332, Alé, 2013f, report JSM0480]. Two knapsack or boom-spray applications (24.8–26.8 g ai/ha) with an interval of 13–14 days were applied at in a volume of 243–313 L/ha with the last treatment at BBCH 41–49. Trials were performed in North and South Europe and include four decline trials. The results are summarised in Table 72.

Table 72 Supervised field trials on cauliflower treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

CAULI-FLOWER Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Jeinsen, Germany, 2012 (Faraday)	2 (14)	26.1 24.8	10 10	BBHC 43-49 Oct 10	0 3 7 14 21	< 0.01/0.01 (0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/0.02 (0.02) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0332; JSM0332-01; [Alé E, 2013e]
Innenheim, France (North), 2012 (Korlanu)	2 (14)	26.7 26.3	10 10	BBCH 41-43 Oct 01	0 3 7 15 21	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0332; JSM0332-02; [Alé E, 2013e]
Ven-Zelderheide, The Netherlands, 2012 (Oviedo)	2 (14)	25.3 25.9	8 8	BBCH 43-49 Aug 30	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0332; JSM0332-03; [Alé E, 2013e]
Innenheim, France (North), 2013 (Hermon)	2 (14)	26.7 26.4	10 10	BBCH 41-43 June 11	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0480; JSM0480-01; [Alé E, 2013f]
Mondonville, France (South), 2012 (Romanesco)	2 (14)	26.8 26.5	10 10	BBCH 43 Oct 23	0 3 7 14 21	0.03/0.04 (0.04) 0.03 0.01 0.01/< 0.01 (0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.04/ 0.05 (0.05) 0.04 0.02 0.02/< 0.01 (0.02) < 0.01	JSM0332; JSM0332-04; [Alé E, 2013e]
Il Secco, Italy, 2012 (Passion)	2 (14)	25.4 24.9	10 10	BBCH 45 Oct 14	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0332; JSM0332-05; [Alé E, 2013e]
Canjayar, Spain, 2012 (Hollis)	2 (14)	26.0 25.9	10 10	BBCH 41 Nov 23	0 3 8 15 22	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0332; JSM0332-06; [Alé E, 2013e]
Castelnuovo Scrivia, Italy, 2013 (Oceano)	2 (14)	26.6 26.1	8 8	BBCH 41 Sept 19	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0480; JSM0480-02; [Alé E, 2013f]

Head brassicas

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue trials on head cabbages [Alé, 2013g, report JSM0334, Alé, 2013h, report JSM0482]. Two spray applications (24–30 g ai/ha) with an interval of 13–16 days were applied at in a volume of

240–533 L/ha with the last treatment at BBCH 43–49. Trials were performed in North and South Europe and include six decline trials and some included plots for processing. The results are summarised in Table 73.

Table 73 Supervised field trials on head cabbages treated with a pre-harvest broadcast spray formulation with cyclaniliprole (50SL)

HEAD CABBAGES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Jeinsen, Germany, 2012 (Robustor)	2 (15)	27.3 26.8	10 10	BBCH 44-45 Oct 16	0 3 7 13 21	< 0.01/< 0.01 (< 0.01) 0.02 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) 0.03 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0334; JSM0334-01; [Alé E, 2013g]
Arnum, Germany, 2012 (Lenox)	2 (14)	27.3 27.9	10 10	BBCH 47-48 Sept 04	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0334; JSM0334-02; [Alé E, 2013g]
Donnelay, France (North), 2012 (Milan de pontoise)	2 (14)	27.2 27.5	5 10	BBCH 47 Nov 01	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0334; JSM0334-03; [Alé E, 2013g]
Oberschaeffolsheim, France (North), 2012 (Zerlina)	2 (13)	26.5 26.1	10 10	BBCH 47 Sept 12	0 3 7 15 21	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0334; JSM0334-04; [Alé E, 2013g]
St. Sylvain d`Ánjou, France (North), 2012 (Virosa)	2 (14)	24.7 26.7	10 10	BBCH 44 Oct 01	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0334; JSM0334-05; [Alé E, 2013g]
NW Ebrington, United Kingdom, 2012 (Amazon)	2 (16)	27.0 27.9	10 10	BBCH 46-48 Sept 27	0 3 7 13 21	0.08/0.08 (0.08) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.09/ 0.09 (0.09) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0334; JSM0334-06; [Alé E, 2013g]
Arnum, Germany, 2013 (Balbro)	2 (14)	29.5 25.8	10 10	BBCH 47-49 June 25	0 3 7 14 21	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0482; JSM0482-01; [Alé E, 2013h]
Innenheim, France (North), 2013 (Farao)	2 (14)	24.5 26.0	10 10	BBCH 43 June 11	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0482; JSM0482-02; [Alé E, 2013h]
Mondonville, France (South), 2012 (Firensa)	2 (14)	27.5 26.8	10 10	BBCH 45 Nov 06	0 3 7 14 21	0.03/0.03 (0.03) 0.02 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.04/ 0.04 (0.04) 0.03 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0334; JSM0334-07; [Alé E, 2013g]

HEAD CABBAGES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg[a]			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Mondonville, France (South), 2013 (Capriccio)	2 (14)	26.0 26.1	10 10	BBCH 43 Aug 23	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0482; JSM0482-03; [Alé E, 2013h]
Il Secco, Italy, 2012 (Crespo F1)	2 (14)	25.8 24.9	10 10	BBCH 46 Oct 14	15	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0334; JSM0334-08; [Alé E, 2013g]
Torroella de Montgri – L`Estartit, Spain, 2013 (Savoi Price)	2 (14)	27.4 26.2	10 10	BBCH 43 March 05	0 3 7 14 21	0.06/0.06 (0.06) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.07/ 0.07 (0.07) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0482; JSM0482-04; [Alé E, 2013h]

[a] For some time points (0 and 14 DALT) duplicate field samples were taken. Results are presented as single values and (mean) of duplicate field samples.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on head cabbages in field residue trials in USA and Canada [Wiedmann & McDonald, 2014b, report IB-2013-JLW-028-01-01]. Three spray applications of 61–102 g ai/ha with an interval of 6–8 days were applied at in a volume of 195–290 L/ha at BBCH 47–49 or heads starting to form (BBCH41). At two sites an adjuvant was added to the mix. Trials were performed in 2012 and 2013 growing seasons and include one decline trial. The results are summarised in Table 74.

Table 74 Supervised field trials on cabbage treated with a formulation with cyclaniliprole (50SL) using a pre-harvest back pack or tractor mounted side boom sprayer

CABBAGES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a]			Report; Trial no [ref]
						Parent [a]	NK-1375 [a]	Tot. [a]	
North Rose, NY, USA, 2012 (Storage No. 4)	3 (7,7)	64 62 62	29 29 29	20-54 cm heads Oct 10	1	0.29/0.35 (0.32)	0.018/0.021 (0.020)	0.31/ 0.37 (0.34)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-01 [Wiedmann &McDonald2014b]
Mebane, NC, USA, 2012 (Early Jersy Wakefield)	3 (7,7)	80 78 87	28 29 30	BBCH 49 June 18	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-02 [Wiedmann &McDonald2014b]
Bradenton, FL USA, 2012 (Bravo)	3 (7,7)	68 67 69	24 24 24	heads formed Dec 16	1	0.022/0.031 (0.027)	< 0.01/< 0.01 (< 0.01)	0.033/ 0.042 (0.038)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-03 [Wiedmann &McDonald2014b]
Verona, WI, USA, 2013	3 (7,8)	81 80 83	31 32 32	BBCH 47-49 Aug 23	1	0.016/0.012 (0.014)	< 0.01/< 0.01 (< 0.01)	0.027/ 0.023 (0.025)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-05

CABBAGES Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg [a]			Report; Trial no [ref]
						Parent [a]	NK-1375 [a]	Tot. [a]	
(Bobcat)									[Wiedmann &McDonald2014b]
Seymour, IL, USA, 2013 (Golden acre)	3 (6,8)	76 82 77	28 28 28	BBCH 49 July 23	1	0.071/0.093 (0.082)	< 0.01/ 0.011 (0.011)	0.082/0.10 (0.094)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-06 [Wiedmann &McDonald2014b]
Geneva, MN USA, 2013 (Golden cross)	3 (7,7)	99 99 102	40 39 40	heads > 5-11 cm [a] July 19	1	0.053/0.026 (0.040)	0.01/ < 0.01 (0.01)	0.063/ 0.037 (0.051)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-07 [Wiedmann &McDonald2014b]
Bagley, IA, USA, 2013 (Stonehead)	3 (7,8)	80 79 78	39 38 36	BBCH 47 July 12	0 1 3 7	0.36/0.32 (0.34) 0.11/0.19 (0.15) 0.14/0.13 (0.13) 0.032/0.065 (0.049)	0.027/0.018 (0.023) 0.014/0.023 (0.019) 0.025/0.021 (0.023) 0.016/0.013 (0.015)	0.39/ 0.34 (0.36) 0.12/ 0.21 (0.17) 0.16/ 0.15 (0.16) 0.049/ 0.079 (0.065)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-08 [Wiedmann &McDonald2014b]
Hinton, OK, USA, 2013 (Late Flat Dutch) + [ADJ]	3 (6,7)	82 93 89	42 46 43	BBCH 49 June 20	1	0.032/0.017 (0.025)	< 0.01/< 0.01 (< 0.01)	0.042/ 0.027 (0.035)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-09 [Wiedmann &McDonald2014b]
Madera, CA, USA, 2012 (Golden Cross) + [ADJ]	3 (7,7)	61 61 61	22 21 21	mature cabbage heads, Dec 27	1	0.40/0.39 (0.39)	0.031/0.025 (0.028)	0.43/ 0.41 (0.42)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-10 [Wiedmann &McDonald2014b]
St-Marc-sur-Richelieu, QC CANADA 2013 (Bronco)	3 (7,7)	84 82 79	40 40 40	BBCH 47-49 July 25	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-04 [Wiedmann &McDonald2014b]

[ADJ] = Adjuvant (Baron (site 09) or R-11 (site 10) Adjuvant) was added with application.

[a] = reported growth stage (vegetative) not correct, because growth stage at second application the heads were already 5–11 cm.

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue trials on Brussels sprouts [Alé, 2013c, report JSM0340, Alé, 2013d, report JSM0484, Alé 2014, report JSM0603]. Two ground directed boomspray applications (25–28 g ai/ha) with an interval of 14–15 days were applied at in a volume of 243–311 L/ha at BBCH 43–47, apart from one trial (JSM0340-03) where the last treatment was applied at BBCH 88–92. Trials were performed in North and South Europe and include four decline trials. The results are summarised in Table 75.

Table 75 Supervised field trials on Brussels sprouts treated with a pre-harvest broadcast spray formulation with cyclaniliprole (50SL)

BRUSSELS SPROUTS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Jeinsen, Germany, 2012 (Maximus)	2 (14)	26.8 26.3	10 10	BBCH 45-47 Nov 31	14	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	JSM0340; JSM0340-01; [Alé E, 2013c]
Donnelay, France (North), 2012 (Sandra)	2 (14)	27.2 27.8	10 10	BBCH 45 Nov 01	0 3 8 14 21	0.02/0.02 (0.02) 0.02 0.02 0.02/0.02 (0.02) 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.03/ 0.03 (0.03) 0.03 0.03 0.03/ 0.03 (0.03) 0.01	JSM0340; JSM0340-02; [Alé E, 2013c]
NW Ebrington, United Kingdom, 2012 (Crispus)	2 (15)	25.4 24.8	10 10	BBCH 88-92 Nov 14	0 3 8 14 21	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0340; JSM0340-03; [Alé E, 2013c]
Meterik, The Netherlands, 2012 (Abacus)	2 (15)	25.9 26.1	8 8	BBCH 47 Aug 27	13	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0484; JSM0484-01; [Alé E, 2013d]
Mondonville, France (South), 2013 (Cronus)	2 (14)	26.0 25.8	10 10	BBCH 43 Sept 06	0 3 7 14 21	0.02/0.02 (0.02) 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.03/ 0.03 (0.03) 0.02 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	JSM0484; JSM0484-02; [Alé E, 2013d]
Abelouzos-Kapariana, Greece, 2013 (Franklin F1)	2 (14)	27.1 25.3	8 8	BBCH 47 Aug 28	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JSM0484; JSM0484-05; [Alé E, 2013d]
Castelnuovo Scrivia, Italy, 2013 (Exodus)	2 (14)	25.2 26.3	8 8	BBHC 46 Nov 20	0 2 7 14 21	0.05/0.06 (0.06) 0.03 0.03 0.04/0.04 (0.04) 0.03	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01	0.06/0.07 (0.07) 0.04 0.04 0.05/ 0.05 (0.05) 0.04	JSM0603; JSM0603-01; [Alé E, 2014]
Cuneo, Italy, 2013 (Rampart)	2 (14)	26.4 25.3	8 8	BBCH 47 Dec 09	14	0.04/0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.05)	JSM0603; JSM0603-01; [Alé E, 2014]

[GS] it appears that the growth stage is not representative for commercial harvest.

[a] For some time points (0 and 14 DALT) duplicate field samples were taken. Results are presented as single values and (mean) of duplicate field samples.

Fruiting vegetables - Cucurbits

Cucumber

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in field residue trials on cucumbers in the USA and in Canada [Wiedmann and McDonald, 2014b, report IB-2013-JAM-003-01]. Three spray applications at 76–84 g ai/ha, with an interval of 6–8 days were applied with spray volumes ranging from 183-375 L/ha using CO_2 backpack sprayers or tractor mounted boom sprayers. Except for trial IB-2013-JAM-003-02 an adjuvant was added to the mix. Trials were performed in the growing season 2013 and include one decline trial. Sample included fruits from ≥ 12 plants and/or weighed more than 2 kg unless indicated differently [SS]. The results are summarised in Table 76.

Table 76 Supervised field trials on cucumber treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

CUCUMBER Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Seven Springs, NC, USA, 2013 (National Pickling) + [ADJ]	3 (7-8)	80 80 79	27 25 25	BBCH 89 June 19	1	0.028/0.020 (0.024)	< 0.01/< 0.01 (< 0.01)	0.039/ 0.031 (0.035)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-01; [Wiedmann & McDonald, 2014bc]
Suffolk, VA, USA, 2013 (Straight Eight)	3 (7)	82 83 84	34 35 35	0.9-1.5 m runners July 19	1	0.018/0.019 (0.019)	< 0.01/< 0.01 (< 0.01)	0.029/0.030 (0.030)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-02; [Wiedmann & McDonald, 2014b]
Winter Garden, FL, USA, 2013 (BOA) + [ADJ]	3 (7)	80 79 78	21 21 21	BBCH 83 June 05	1	< 0.01/< 0.01 (< 0.01) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	IB-2013-JAM-003-01; IB-2013-JAM-003-01-03; [Wiedmann & McDonald, 2014b]
Northwood, ND, USA, 2013 (Marketmore 76)	3 (7)	81 81 81	28 28 28	BBCH 85 Aug 22	1	0.021/0.015(0.018)	< 0.01/< 0.01 (< 0.01)	0.032/ 0.026 (0.029)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-05; [Wiedmann & McDonald, 2014b]
Seymour, IL, USA, 2013 (Bush Crop F1)	3 (7)	83 76 80	28 28 28	BBCH 87 Aug 08	1	0.016/0.012 (0.014)	< 0.01/< 0.01 (< 0.01)	0.027/ 0.023 (0.025)	IB-2013-JAM-003-01; IB-2013-

CUCUMBER Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
+ [ADJ]									JAM-003-01-06; [Wiedmann & McDonald, 2014b]
Enid, OK USA, 2013 (Calypso-pickle) + [ADJ]	3 (6-7)	77 79 81	42 41 32	BBCH 88 Aug 14	1	< 0.01/0.016 (0.013)	< 0.01/< 0.01 (< 0.01)	< 0.01/ 0.027 (0.024)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-07; [Wiedmann & McDonald, 2014b]
Leonard, MO, USA, 2013 (Jackson Classix, F1) + [ADJ]	3 (7)	82 82 82	30 29 29	flower to ripe fruit Aug 19	0 1 4 7	0.049/0.061 (0.055) 0.025/0.025 (0.025) 0.015/0.016 (0.016) 0.015/0.010 (0.013)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.060/ 0.072 (0.066) 0.036/ 0.036 (0.036) 0.026/ 0.027 (0.027) 0.026/ 0.021 (0.024)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-08; [Wiedmann & McDonald, 2014b]
Uvalde, TX, USA, 2013 (Cobra F1) + [ADJ]	3 (7)	79 81 82	38 40 37	BBCH 81 Aug 30	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-09; [Wiedmann & McDonald, 2014b]
St-Marc-sur-Richelieu, Quebec, CAN, 2013 (Marketmore) + [ADJ]	3 (6-7)	83 81 80	40 40 40	BBCH 67-71 July 31	1	0.010/0.012 (0.011)	< 0.01/< 0.01 (< 0.01)	0.021/ 0.023 (0.022)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-04; [Wiedmann & McDonald, 2014b]

[ADJ] = Adjuvant (NIS = non-ionic surfactant) was added with application.

[SS] = sample size weighed less than the required 2 kg (1.36 and 1.81 kg for both field samples), with at least 12 fruits picked from at least 12 plants/locations on the plot. Samples were considered suitable enough to be used for maximum residue estimation.

Summer squash

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in field residue trials on summer squash in the USA [Wiedmann and McDonald, 2014b, report IB-2013-JAM-003-01]. Three spray applications at 77–83 g ai/ha, with an interval of 6–8 days were applied with tractor mounted boomsprayer of knapsack sprayers using spray volumes ranging from 183–378 L/ha.

Except for trial IB-2013-JAM-003-10 an adjuvant was added to the mix. Trials were performed in the growing season 2013 and include one decline trial. The results are summarised in Table 77.

Table 77 Supervised field trials on summer squash treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

SUMMER SQUASH Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Germansville, PA, USA, 2013 (Super Pik)	3 (6)	81 80 80	34 34 34	full boom, fruiting, mature fruit Aug 11	1	0.028/0.029 (0.028)	< 0.01/< 0.01 (< 0.01)	0.039/ 0.039 (0.039)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-10; [Wiedmann & McDonald, 2014bc]
Seven Spring, NC, USA, 2013 (Early Prolific Straight Neck) + [ADJ]	3 (6-8)	79 80 79	28 27 29	BBCH 88 June 10	0 1 4 7	0.043/0.027 (0.035) 0.011/0.017 (0.014) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.054/ 0.038 (0.046) 0.022/ 0.028 (0.025) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-11; [Wiedmann & McDonald, 2014b]
Wintergarden, FL, USA 2013 (Goldstar) + [ADJ]	3 (7)	80 80 81	21 21 21	BBCH 83 June 05	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-12; [Wiedmann & McDonald, 2014b]
Northwood, ND, USA, 2013 (Black Beauty) + [ADJ]	3 (6-8)	78 81 79	28 28 28	fruit have reached typical length and colour Aug 16	1	0.024/0.027 (0.026)	< 0.01/< 0.01 (< 0.01)	0.035/ 0.038 (0.037)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-13; [Wiedmann & McDonald, 2014b]
Seymour, IL, USA, 2013 (Sunglo F1) + [ADJ]	3 (7)	81 81 82	28 28 29	BBCH 88 Aug 08	1	0.040/0.051 (0.046)	< 0.01/< 0.01 (< 0.01)	0.051/ 0.062 (0.057)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-14; [Wiedmann & McDonald, 2014b]
Enid, OK, USA, 2013 (Calabacita) + [ADJ]	3 (7)	79 82 77	37 37 42	BBCH 76 Aug 01	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-15; [Wiedmann & McDonald, 2014b]
Leonard, MO, USA, 2013 (Dunja F1) + [ADJ]	3 (7)	79 80 80	38 40 41	51 cm tall with fruit July 16	1	0.028/0.027(0.028)	0.012/< 0.01 (0.011)	0.041/ 0.038 (0.040)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-16; [Wiedmann & McDonald, 2014b]
Madera, CA, USA, 2013 (Black Beauty) + [ADJ]	3 (7)	81 83 81	29 29 28	Mature summer squash June 20	1	0.031/0.034 (0.033)	< 0.01/< 0.01 (< 0.01)	0.042/ 0.045 (0.043)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-17; [Wiedmann & McDonald, 2014b]

SUMMER SQUASH Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Hillsboro, OR, USA, 2013 (Hybrid Squash – Zukes) + [ADJ]	3 (7)	79 80 78	34 34 34	BBCH 85-88	1	0.014/0.017 (0.016)	< 0.01/< 0.01 (< 0.01)	0.025/ 0.028 (0.027)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-18; [Wiedmann & McDonald, 2014b]

[ADJ] = Adjuvant (NIS = non-ionic surfactant) was added with application.

Melons/Canteloupe

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in field residue trials on and cantaloupes in the USA and in Canada [Wiedmann and McDonald, 2014b, report IB-2013-JAM-003-01]. Three spray applications at 76–85 g ai/ha, with an interval of 6–8 days were applied with spray volumes ranging from 190–321 L/ha using a handheld boomsprayer, tractor mounted boomsprayer or a (CO_2) back sprayer. Except for trial IB-2013-JAM-003-19 and 20 an adjuvant was added to the mix. Trials were performed in the growing season 2013 and include one decline trial. The results are summarised in Table 78.

Table 78 Supervised field trials on melon (Cantaloupe) treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

MELON Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg*			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Jeffersonville, GA, USA, 2013 (Hales Best Jumbo)	3 (7)	82 82 79	29 29 28	BBCH 87 Jul 15	1	0.074/0.100 (0.087)	< 0.01/0.011 (0.011)	0.085/ 0.11 (0.099)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-19; [Wiedmann & McDonald, 2014bc]
Northwood, ND, USA, 2013 (Athena)	3 (7)	82 81 80	28 34 28	BBCH 88 Sep 10	1	0.036/0.042 (0.039)	< 0.01/< 0.01 (< 0.01)	0.047/ 0.053 (0.050)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-20; [Wiedmann & McDonald, 2014b]
Enid, OK, USA, 2013 (CPMR 45) + [ADJ]	3 (7)	80 80 80	31 35 42	BBCH 89 Aug 28	1	0.032/0.070 (0.051)	< 0.01/0.012 (0.011)	0.043/ 0.083 (0.063)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-22; [Wiedmann & McDonald, 2014b]
Seymour, IL, USA, 2013 (Lil'Loupe) + [ADJ]	3 (7)	83 76 80	28 28 29	BBCH 83 Aug 29	1	0.035/< 0.01 (0.023)	< 0.01/< 0.01 (< 0.01)	0.046/ 0.021 (0.033)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-23; [Wiedmann & McDonald, 2014b]

MELON Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg* Parent	NK-1375	Tot.	Report; Trial no [ref]
Leonard, MO, USA, 2013 (SC Earlichamp F1) + [ADJ]	3 (7)	81 81 81	29 29 29	green fruit to ripe fruit Aug 19	1	0.040/0.044 (0.042)	0.012/0.011 (0.012)	0.053/ 0.056 (0.055)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-24; [Wiedmann & McDonald, 2014b]
Hinton, OK, USA, 2013 (Superstar) + [ADJ]	3 (7-8)	81 80 82	43 39 43	BBCH 83 Aug 13	1	0.012/0.015 (0.014)	< 0.01/< 0.01 (< 0.01)	0.023/ 0.026 (0.024)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-25; [Wiedmann & McDonald, 2014b]
Madera, CA, USA, 2013 (Hales Best Jumbo) + [ADJ]	3 (7)	81 82 81	29 29 29	mature cantaloupe July 15	0 1 4 7	0.052/0.057 (0.055) 0.030/0.050 (0.040) 0.019/0.027 (0.023) 0.014/0.012 (0.013)	0.012/0.017 (0.015) 0.014/0.020 (0.017) 0.013/0.018 (0.016) < 0.01/< 0.01 (< 0.01)	0.065/ 0.075 (0.070) 0.045/ 0.071 (0.058) 0.032/ 0.046 (0.040) 0.025/ 0.023 (0.024)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-26; [Wiedmann & McDonald, 2014b]
Sanger, CA, USA, 2013 (Hybrid) + [ADJ]	3 (6-8)	77 83 79	28 28 28	BBCH 89 Oct 08	1	0.041/ 0.047 (0.044)	< 0.01/< 0.01 (< 0.01)	0.052/ 0.058 (0.055)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-27; [Wiedmann & McDonald, 2014b]
Porterville, CA, USA, 2013 (Top Mark) + [ADJ]	3 (7)	80 79 80	28 28 28	BBCH 89 July 25	1	0.013/ 0.021 (0.017)	< 0.01/< 0.01 (< 0.01)	0.024/ 0.032 (0.028)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-28; [Wiedmann & McDonald, 2014b]
Branchton, Ontario, CAN, 2013 (Melon Muskat Hybrid) + [ADJ]	3 (6-7)	85 82 77	27 26 26	BBCH 84-86 Sep 17	1	0.073/ 0.068 (0.071)	< 0.01/< 0.01 (< 0.01)	0.084/ 0.079 (0.081)	IB-2013-JAM-003-01; IB-2013-JAM-003-01-21; [Wiedmann & McDonald, 2014b]

[ADJ] = Adjuvant (NIS = non-ionic surfactant) was added with application.

Fruiting vegetables, other than Cucurbits

Field residue trials were submitted on tomato (outdoor and indoor), sweet peppers (outdoor and indoor) and 1 indoor trial on hot peppers (Korea).

Sweet peppers, outdoor applications

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in European residue outdoor trials on sweet peppers [Alé, 2013i, report JSM0336, Alé, 2013j, report JSM0485l]. Two groundboom or lance spray applications (39-44 g ai/ha) with an interval of 10–11 days were applied at a volume of 369–539 L/ha. Report JSM0036 [Alé, 2013k] was a comparative study, where on one plot of each test site, in addition to the regular treatment, an adjuvant [ADJ] was

applied. Trials were performed in North and South Europe and include 8 decline trialsThe results are summarised in Table 79.

Table 79 Supervised outdoor field trials on sweet peppers treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL) with or without adjuvant [ADJ]

PEPPERS, SWEET Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Gerichshain, Germany, 2012 (Yolo wonder)	2 (10)	41.7 38.5	10 10	BBHC 86 Oct 04	0 1 3 7	0.02/0.02 (0.02) 0.02/0.02 (0.02) 0.02 0.01	< 0.01/< 0.01 < 0.01 < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.03 (0.03) 0.03/0.03 (0.03) 0.03 0.02	JSM0336 JSM0336-01 [Alé E., 2013i]
idem + [ADJ]	idem	40.4 41.1	10 10	idem	0 1 3 7	0.03 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.03 0.03 0.03	idem
Jeinsen, Germany, 2012 (Orange beauty)	2 (10)	41.4 41.4	10 10	BBCH 75-79 Sept 24	1 3	0.02/0.03 (0.02) [SS] 0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01) [SS] < 0.01/< 0.01 (< 0.01)	0.03/0.04 (0.04) [SS] 0.03/0.02 (0.03)	JSM0336 JSM0336-02 [Alé E., 2013i]
idem + [ADJ]	idem	42.4 40.3	10 10	idem	1 3	0.02 [SS] 0.01	< 0.01 [SS] < 0.01	0.03 [SS] 0.02	idem
Handschuheim, France (North), 2012 (Lamuyo F1)	2 (11)	39.2 39.8	10 10	BBCH 87-89 Sept 3	0 1 3 7	0.02/0.02 (0.02) 0.02/0.02 (0.02) 0.02 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.03 (0.03) 0.03/ 0.3 (0.03) 0.03 0.02	JSM0336 JSM0336-03 [Alé E., 2013i]
idem + [ADJ]		41.741.7	10 10	idem	0 1 3 7	0.03 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.03 0.02 0.02	idem
Donnelay, France (North), 2012	2 (10)	44.1 42.1	10 10	BBCH 86 Sept 06	1 3	0.02/0.02 (0.02) < 0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03) < 0.01/ 0.02 (0.02)	JSM0336 JSM0336-04 [Alé E., 2013i]
idem + [ADJ]		44.1 44.1	10 10	idem	1 3	0.02 0.02	< 0.01 < 0.01	0.03 0.03	idem
Szegvár, Hungary, 2013 (Bihar)	2 (11)	42.1 40.4	10 10	BBCH 86 Aug 27	0 1 3 7	0.02/0.01 (0.02) 0.01/0.01 (0.01) 0.01 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.02 (0.03) 0.02/ 0.02 (0.02) 0.02 0.02	JSM0485 JSM0485-01 [Alé E., 2013j]
Bugyi, Hungary, 2013 (Kapitex F1)	2 (11)	44.1 39.1	10 10	BBCH 87-89 Aug 27	1 3	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01) < 0.01/ < 0.01 (< 0.01)	JSM0485 JSM0485-02 [Alé E., 2013j]
Wunstorf, Germany, 2013 (Red beauty)	2 (10)	44.143.1	10 10	BBCH 88-89 Sept 16	1 3	0.01/0.02 (0.02) < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.02/ 0.03 (0.03) < 0.01/ < 0.01 (< 0.01)	JSM0485 JSM0485-03 [Alé E., 2013j]
Wielun, Poland,	2 (11)	38.8 42.0	10 10	BBCH 88-89	0	0.04/0.03 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.04 (0.05)	JSM0485 JSM0485-04

PEPPERS, SWEET Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2013 (Etiuda)				Sept 10	1 3 7	0.01/< 0.01 (0.01) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.02/ < 0.01 0.02 < 0.01 < 0.01	[Alé E., 2013j]
Orgueil, France (South), 2012 (Lamuyo F1)	2 (10)	40.7 41.4	10 10	BBCH 87 Aug 13	0 1 3 7	0.01/0.02 (0.02) < 0.01/0.01 (0.01) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.02/ 0.03 (0.03) < 0.01/ 0.02 (0.02) < 0.01 < 0.01	JSM0336 JSM0336-05 [Alé E., 2013i]
idem + [ADJ]	idem	42.3 42.6	10 10	idem	0 1 3 7	0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.01 0.02 < 0.01	idem
Duras, France (South), 2012 (Lamuyo F1)	2 (10)	41.9 42.8	10 10	BBCH 89 Oct 27	0 1	0.04/0.03 (0.04) 0.03/0.03 (0.03)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.05/ 0.04 (0.05) 0.04/0.04 (0.04)	JSM0336 JSM0336-06 [Alé E., 2013i]
idem + [ADJ]	idem	41.2 41.7	10 10	idem	0 1	0.03 0.05	< 0.01 < 0.01	0.041 0.061	idem
Isola Sant Antonio Piemonte, Italy, 2012 (Cuneo)	2 (10)	40.5 41.6	10 10	BBCH (703) [c]	0 1 3 7	0.02/0.01 (0.02) 0.02/0.01 (0.02) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.02 (0.03) 0.03/ 0.02 (0.03) < 0.01 < 0.01	JSM0336 JSM0336-07 [Alé E., 2013i]
idem + [ADJ]	idem	40.7 39.9	10 10	idem	0 1 3 7	0.02 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.03 0.03 < 0.01	idem
Mureio, Italy, 2012 (Cuneo)	2 (11)	41.0 40.2	8 8	BBCH 83 Sept 21	1 3	< 0.01/0.01 (0.01) < 0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/ 0.02 (0.02) < 0.01/ 0.02 (0.02)	JSM0336 JSM0336-08 [Alé E., 2013i]
idem + [ADJ]	idem	41.0 36.9	8 8	idem	1 3	0.02 0.01	< 0.01 < 0.01	0.03 0.02	idem
Santa Susana, Spain, 2012 (Ital rest)	2 (10)	40.7 40.7	10 10	BBCH 71-85 Sept 03	0 1 3 7	0.08/0.05 (0.07) 0.02/0.03 (0.03) 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.09/ 0.06 (0.08) 0.03/0.04 (0.04) 0.02 < 0.01	JSM0336 JSM0336-09 [Alé E., 2013i]
idem + [ADJ]	idem	40.4 41.4	10 10	idem	0 1 3 7	0.08 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.09 0.04 0.03 0.03	idem
Kastana, Greece, 2012 (Florinis)	2 (11)	37.6 40.7	10 10	BBCH 75 Sept 17	1 3	0.02/< 0.01 (0.02) 0.02/< 0.01 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/< 0.01 (0.03) 0.03/ < 0.01 (0.03)	JSM0336 JSM0336-10 [Alé E., 2013i]
idem	idem	43.9 37.6	10 10	idem	1 3	0.02 0.02	< 0.01 < 0.01	0.03 0.03	idem

PEPPERS, SWEET Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
+ [ADJ]									
Isola Sant Antonio Piemonte, Italy, 2013 (Cuneo)	2 (10)	40.9 43.9	8 8	BBCH 87 Aug 23	0 1 3 8	0.02/0.02 (0.02) 0.01/0.01 (0.01) 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.03 (0.03) 0.02/ 0.02 (0.02) 0.02 < 0.01	JSM0485 JSM0485-05 [Alé E., 2013j]
Santa Susana, Spain, 2013 (Ital rest)	2 (11)	38.7 42.1	8 8	BBCH 65-85 Aug 13	1 3	0.05/0.03 (0.04) 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.06/ 0.04 (0.05) 0.03/ 0.03 (0.03)	JSM0485 JSM0485-06 [Alé E., 2013j]

[SS] sample size below the require 2 kg (1.0-1.25 kg)

[ADJ] = commercial adjuvant codacide, oil seed rape oil 95%

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on bell peppers and non-bell peppers in field residue trials in USA (8 and 3, respectively) and Canada (one on bell pepper) [Wiedmann & McDonald, report IB-2012-JLW-029-01-01]. Three spray applications of 60–83 g ai/ha with an interval of 6–8 days were applied at in a volume of 200–379 L/ha. An adjuvant was added to the mix at one test site (32). Trials were performed in 2012 and 2013 growing seasons and included one decline trial. Samples consisted of fruits from at least 12 plants and/or weighed > 1 kg and were picked at mature stage. Only the samples picked in trials 26, 27 and 31 weighed > 2 kg, the other samples weighed 1.1–1.7 kg. The results are summarised in Table 80 and Table 81.

Table 80 Supervised field trials on sweet bell peppers treated with a formulation with cyclaniliprole (50SL) using a pre-harvest knapsack sprayer or tractor mounted boom sprayer

BELL PEPPERS, SWEET Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Seven Springs, NC, USA, 2013 (California Wonder)	3 (7,7)	82 79 81	25 25 24	BBCH 89 June 26	1	0.019/ 0.019 (0.019) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.029/ 0.029 (0.029) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-24 [Wiedmann &McDonald2014c]
Winter Garden, FL, USA, 2013 (New Ace)	3 (6,7)	81 81 80	21 21 21	BBCH 82 June 18	1	0.088/ 0.11 (0.098) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.099/ 0.12 (0.11) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-25 [Wiedmann &McDonald2014c]
Northwood, ND, USA, 2013 (Bell)	3 (6,8)	78 81 80	28 29 28	fruit reached final length and colour Aug 16	1	0.048/ 0.048 (0.048)	< 0.01/< 0.01 (< 0.01)	0.059/ 0.059 (0.059)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-27 [Wiedmann &McDonald2014c]
Seymour,	3	82	29	BBCH	1	0.070/	< 0.01/< 0.01	0.080/	IB-2012-JLW-029-

BELL PEPPERS, SWEET Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
IL, USA, 2013 (King Arthur F1)	(7,7)	77 79	28 29	84 Sept 24		0.074 (0.072) [SS]	(< 0.01) [SS]	0.085 (0.083) [SS]	01-01 IB-2012-JLW-029-01-01-28 [Wiedmann &McDonald2014c]
Geneva, MN, USA, 2013 (California Wonder)	3 (6,8)	80 83 82	40 41 38	fruiting Sept 23	1	0.028/ 0.021 (0.025) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.039/ 0.031 (0.035) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-29 [Wiedmann &McDonald2014c]
Uvalde TX, USA, 2012 (Taurus)	3 (7,7)	62 60 61	26 26 26	BBCH 84 Sept 25	1	0.045/ 0.047 (0.046) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.055/ 0.058 (0.056) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-30 [Wiedmann &McDonald2014c]
Sanger, CA, USA, 2012 (Indra)	3 (7,7)	60 60 61	16 16 16	BBCH 89 Oct 18	1	0.098 / 0.10 (0.10)	0.016/0.019 (0.018)	0.11/ 0.12 (0.12)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-31 [Wiedmann &McDonald2014c]
Madera, CA, USA, 2013 (Cypress) + [ADJ]	3 (7,7)	81 82 81	29 28 29	mature (medium to large) June 28	0 1 3 7	0.10/ 0.07 (0.086) [SS] 0.077/ 0.060 (0.068) [SS] 0.038/ 0.037 (0.037) [SS] 0.035/ 0.032 (0.033) [SS]	0.023/ 0.018 (0.020) [SS] 0.028/ 0.024 (0.026) [SS] 0.018/ 0.020 (0.019) [SS] 0.027/ 0.031 (0.029) [SS]	0.13/ 0.089 (0.11) [SS] 0.11/ 0.085 (0.096) [SS] 0.057/ 0.063 (0.058) [SS] 0.064/ 0.065 (0.064) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-32 [Wiedmann &McDonald2014c]
St-Marc-sur-Richelieu, Quebec, CANADA, 2013 (Joker)	3 (6,8)	75 80 75	32 32 32	BBCH 71-73 Aug 22	1	0.015/ 0.013 (0.014)	< 0.01/< 0.01 (< 0.01)	0.025/ 0.024 (0.025)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-26 [Wiedmann &McDonald2014c]

[ADJ] = Adjuvant (R-11 Adjuvant (site 32)) was added with application.

[SS] = sample size below the required 2 kg, but >1 kg, with at least 12 fruits picked from at least 12 plants/locations on the plot. Samples were considered suitable enough to be used for maximum residue estimation.

Table 81 Supervised field trials on non-bell sweet peppers treated with a formulation with cyclaniliprole (50SL) using a pre-harvest backpack sprayer or tractor mounted boom sprayer

NON-BELL	No,	g	g	GS &	DALT	Residues, mg/kg	Report;

PEPPERS, SWEET Location, Country; year; (variety)	(interval)	ai/ha	ai/hL	last treatment day	(days)	Parent	NK-1375	Tot.	Trial no [ref]
Winter Garden, FL, USA, 2013 (Calica)	3 (6,7)	80 80 80	21 21 21	BBCH 82 June 18	1	0.057/0.056 (0.057) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.068/ 0.067 (0.067) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-33 [Wiedmann &McDonald2014c]
Seymour, IL, USA, 2013 (Sopron F1)	3 (7,8)	81 77 77	29 29 29	BBCH 89 Sept 03	1	0.036/0.046 (0.041) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.046/ 0.056 (0.051) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-34 [Wiedmann &McDonald2014c]
Porterville, CA, USA, 2013 (Mammonth)	3 (7,7)	80 80 80	28 28 28	BBCH 89 July 25	1	0.074/0.080 (0.077) [SS]	0.019/0.013 (0.016) [SS]	0.094/ 0.095 (0.094) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-35 [Wiedmann &McDonald2014c]

[SS] = sample size below the required 2 kg, but >1 kg, with at least 12 fruits picked from at least 12 plants/locations on the plot. Samples were considered suitable enough to be used for maximum residue estimation.

Sweet pepper, indoor applications

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in eight European residue trials on sweet peppers [Alé, 2013k, report JSM0337, Alé, 2013l, report JSM0487]. Two boom or lancespray applications (37.3–44.4 g ai/ha) with an interval of 6-8 days were applied at a volume of 914–1090 L/ha with the last application at BBCH 27–89. Trials were performed in glasshouses in North and South Europe and include four decline trials. Report JSM00337 [Alé, 2013k] was a comparative study, where on one plot of each test site, in addition to the regular treatment, an adjuvant [ADJ] was applied. Samples consisted of at least 12 fruits without stems weighing more than 2 kg, unless indicated otherwise [SS]. The results are summarised in Table 82.

Table 82 Supervised indoor greenhouse trials on peppers treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL) with or without an adjuvant [ADJ]

PEPPERS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Belfield, the Netherlands, 2012 (Nagano)	2 (6)	41.8 38.2	4 4	BBHC 87-89 Oct 16	1 3	0.02/0.03 (0.03) 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/0.04 (0.04) 0.03/0.03 (0.03)	JSM0337 JSM0337-01 [Alé E., 2013k]
idem +[ADJ]	idem	41.7 42.6	4 4	idem	1 3	0.02 0.03	< 0.01 < 0.01	0.03 0.04	idem
Meterik, the Netherlands, 2012 (Viper)	2 (7)	40.0 38.5	4 4	BBCH 87-89 Oct 22	0 1 3 7	0.02/0.02 (0.02) 0.03/0.02 (0.03) 0.02 0.02	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/0.03 (0.03) 0.04/0.03 (0.04) 0.03 0.03	JSM0337 JSM0337-02 [Alé E., 2013k]
idem + [ADJ]	idem	41.3 38.6	4 4	idem	0 1 3 7	0.02 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.03 0.03 0.02	idem

PEPPERS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Meterik, the Netherlands, 2013 (Viper)	2 (8)	41.4 42.1	4 4	BBCH 87-89 June 29	1 3	0.04/0.04 0.04 0.03/0.04 (0.04)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.05/0.05 (0.05) 0.04/0.05 (0.05)	JSM0487 JSM0487-01 [Alé E., 2013l]
Duras, France (South), 2012 (Lamuyo F1)	2 (7)	37.3 41.3	4 4	BBCH 89 Oct 22	0 1 3 7	0.05/0.04 0.05 [SS] 0.02/0.04 (0.03) [SS] 0.03 [SS] 0.02 [SS]	< 0.01/< 0.01 (< 0.01) [SS] < 0.01/< 0.01 (< 0.01) [SS] < 0.01 [SS] < 0.01 [SS]	0.06/0.05 (0.06) [SS] 0.03/0.05 (0.04) [SS] 0.04 [SS] 0.03 [SS]	JSM0337 JSM0337-03 [Alé E., 2013k]
idem + [ADJ]	idem	38.8 40.7	4 4	idem	0 1 3 7	0.05 [SS] 0.04 [SS] 0.04 [SS] 0.03 [SS]	< 0.01 [SS] < 0.01 [SS] < 0.01 [SS] < 0.01 [SS]	0.06 [SS] 0.05 [SS] 0.05 [SS] 0.04 [SS]	idem
Carmagnola, Italy, 2012 (Stellor)	2 (7)	41.0 42.8	4 4	BBCH 87 Oct 8	0 1 3 7	0.04/0.05 (0.05) 0.03/0.03 (0.03) 0.04 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.05/0.06 (0.06) 0.04/0.04 (0.04) 0.05 0.02	JSM0337 JSM0337-04 [Alé E., 2013k]
idem + [ADJ]	idem	44.4 43.2	4 4	idem	0 1 3 7	0.03 0.03 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.04 0.04 0.03	idem
Kapariana, Greece, 2012 (Velisa)	2 (7)	43.0 42.8	4 4	BBCH (703)[a]	1 3	0.04/0.05 (0.05) 0.04/0.03 (0.04)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.05/0.06 (0.06) 0.05/0.04 (0.05)	JSM0337 JSM0337-05 [Alé E., 2013k]
idem + [ADJ]	idem	41.1 40.3	4 4	idem	1 3	0.03 0.03	< 0.01 < 0.01	0.041 0.041	idem
Stenimachos, Greece, 2012 (Banan)	2 (7)	37.7 37.7	4 4	BBCH 72 Aug 30	1 3	0.04/0.04 (0.04) 0.01/0.03 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.05/0.05 (0.05) 0.02/0.04 (0.03)	JSM0337 JSM0337-06 [Alé E., 2013k]
idem + [ADJ]	idem	38.4 38.7	4 4	idem	1 3	0.04 0.03	< 0.01 < 0.01	0.05 0.04	idem
El Ejido, Spain, 2013 (Magno)	2 (6)	40.1 39.7	4 4	BBCH 87 March 31	0 1 3 7	0.04/0.05 (0.05) 0.02/0.03 (0.03) 0.02 0.02	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.05/0.06 (0.06) 0.03/0.04 (0.04) 0.03 0.03	JSM0487 JSM0487-02 [Alé E., 2013l]

[ADJ] = commercial adjuvant codacide, oil seed rape oil 95%

[SS] Samples sizes were al below 2 kg at all sampling intervals, but included 12 fruits.

[a] 703 = 3rd fruit has reached typical size and form (= BBCH 73)

Chili pepper, indoor application

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 4.5%SL) was applied in red pepper, under greenhouse conditions on one location in Korea [Cho B., 2013, report no code]. Two plots were sprayed with one or two applications at a rate of 1 or 2×45 g ai/ha with an interval of 7 days at a volume of 2000 L/ha. The results are summarised in Table 83.

Table 83 Supervised outdoor field trials on chili peppers treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL) with or without adjuvant

CHILI PEPPER Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375[a]	Tot.	
1766 Pyungchun-ri, Sangnam-myun, KOREA, 2013 (Nokkwang)	1	45	2	nr March 29	0	0.24/ 0.22/ 0.23 (0.23)	< 0.01	0.25/0.23/ 0.24 (0.24)	JSM0335 JSM0335-03 [Alé E., 2013p]
					1	0.27/ 0.19/ 0.26 (0.24)	< 0.01	0.28/0.20/ 0.27 (0.25)	
					3	0.22/ 0.23/ 0.22 (0.22)	< 0.01	0.23/0.24/ 0.23 (0.23)	
					5	0.20/ 0.22/ 0.21 (0.21)	< 0.01	0.22/0.23/ 0.22 (0.23)	
					7	0.11/ 0.08/ 0.10 (0.1)	< 0.01	0.12/0.08/ 0.11 (0.11)	
idem	2 (7)	45 45	22	nr March 29	0	0.39/ 0.36/ 0.37 (0.37)	< 0.01	0.40/0.37/ 0.38 (0.38)	idem
					1	0.39/ 0.40/ 0.41 (0.40)	< 0.01	0.40/0.41/ 0.42 (0.41)	
					3	0.36/ 0.38/ 0.36 (0.37)	< 0.01	037/0.39/ 0.37 (0.38)	
					5	0.21/ 0.29/ 0.22 (0.22)	< 0.01	0.22/0.30/ 0.23 (0.23)	
					7	0.16/ 0.11/ 0.14 (0.14)	< 0.01	0.17/0.12/ 0.15 (0.15)	

[a] Since all values were < 0.01, only the means of the triplicate analyses are listed.

Tomato, outdoor applications

A 50 g/L soluble concentrate (IKI-3106 50 SL aka IBE 4064) was applied in 16 European residue outdoor trials on tomatoes [Alé, 2013m, report JSM0335, Alé, 2013n, report JSM0486]. In 8 trials performed in 2012 an additional plot was included using an adjuvant in addition to the regular application. Two groundboom or lance spray applications (37.9–44.8 g ai/ha) with an interval of 10–11 days (except one trial with RTI of 7 days and one trial with RTI of 12 days) were applied at a volume of 319–521 L/ha. Trials were performed in North and South Europe and include 8 decline trials. Report JSM00335 [Alé, 2013p] was a comparative study, where on one plot of each test site, in addition to the regular treatment, an adjuvant [ADJ] was applied. The results are summarised in Table 84.

Table 84 Supervised outdoor field trials on tomatoes treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL) with or without adjuvant

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Thorée les pins, France (North), 2012 (Roma)	2 (7)	41.4 43.5	10 10	BBCH 86 Aug 25	0	/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	JSM0335 JSM0335-03 [Alé E., 2013m]
					1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01)	
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
idem + [ADJ]	idem	42.8 40.7	10 10	idem	0	0.02	< 0.01	0.03	idem
					1	0.01	< 0.01	0.02	
					3	< 0.01	< 0.01	< 0.01	
					7	< 0.01	< 0.01	< 0.01	
Donnelay, France (North), 2012 (Kalimba)	2 (11)	40.1 42.1	10 10	BBCH 89 Aug 10	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01)	JSM0335 JSM0335-04 [Alé E., 2013m]
					3	0.01/0.05 (0.03)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.06 (0.04)	

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg Parent	NK-1375	Tot.	Report; Trial no [ref]
idem + [ADJ]	idem	44.1 44.5	10 10	idem	1 3	0.03 0.02	< 0.01 < 0.01	0.04 0.03	idem
Szegvár, Hungary, 2013 (Uno)	2 (11)	40.639.6	10 10	BBCH 81 Aug 27	0 1 3 7	0.01/0.01 (0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.02/ 0.02 (0.02) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	JSM0486 JSM0486-01 [Alé E, 2013n]
Lajostany, Hungary 2013, (Mobil)	2 (10)	42.6 44.2	10 10	BBCH 87 Sept 06	1 3	0.02/0.02 (0.02) < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/0.03 (0.03) < 0.01/< 0.01 (< 0.01)	JSM0486 JSM0486-02 [Alé E, 2013n]
Goch, Germany, 2013 (Vitella)	2 (10)	42.5 41.2	8 8	BBCH 87-89 Aug 30	0 1 3 7	0.03 /0.02 (0.03) 0.02/0.02 (0.02) 0.02 0.02	<< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.04/ 0.03 (0.04) 0.03/ 0.03 (0.03) 0.03 0.03	JSM0486 JSM0486-03 [Alé E, 2013n]
Wunstorf, Germany, 2013 (Mammouth	2 (10)	43.3 44.3	14 14	BBCH 87-89 Sept 16	1 3	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01) < 0.01/ < 0.01 (< 0.01)	JSM0486 JSM0486-04 [Alé E, 2013n]
Katy Wroclawskie, Poland, 2013 (Zyska)	2 (10)	42.0 41.8	10 10	BBCH 86-89 Sept 09	0 1 3 7	0.03/0.04 (0.04) 0.02/0.02 (0.02) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.04/ 0.05 (0.05) 0.03/ 0.03 (0.03) < 0.01 < 0.01	JSM0486 JSM0486-05 [Alé E, 2013n]
Zabkowice Slaskie, Poland, 2013 (Mieszko)	2 (10)	42.6 41.5	10 10	BBCH 86-89 Sept 09	1 3	0.02/0.02 (0.02) < 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03) < 0.01/ < 0.01 (< 0.01)	JSM0486 JSM0486-06 [Alé E, 2013n]
Duras, France (South), 2012 (Rio grande)	2 (11)	43.5 41.4	10 10	BBCH 89 Sept 30	1 3	0.02/0.01 (0.02) < 0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/ 0.02 (0.03) < 0.01/ < 0.01 (< 0.01)	JSM0335 JSM0335-05 [Alé E., 2013m]
idem + [ADJ]	idem	42.1 40.4	10 10	idem	1 3	0.02 0.01	< 0.01 < 0.01	0.03 0.02	idem
Caderousse, France (South), 2012 (Perfect)	2 (12)	40.744.7	10 10	BBCH 87 Sept 04	0 1 3 7	0.04/0.04 (0.04) 0.02/0.03 (0.03) 0.01 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.05/ 0.05 (0.05) 0.03/ 0.04 (0.04) 0.02 0.02	JSM0335 JSM0335-06 [Alé E., 2013m]
idem + [ADJ]	idem	39.4 44.1	10 10	idem	0 1 3 7	0.04 0.04 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.05 0.05 0.03	idem
Rivarone Piemonte, Italy, 2012 (Nermann)	2 (11)	40.9 40.7	10 10	BBCH 85 Aug 27	1 3	0.02/0.02 (0.02) 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03) 0.03/ 0.03 (0.03)	JSM0335 JSM0335-07 [Alé E., 2013m]
idem + [ADJ]	idem	40.9 39.7	10 10	idem	1 3	0.01 0.01	< 0.01 < 0.01	0.02 0.02	idem
Casei Gerola, Italy,	2 (11)	40.7 44.8	10 10	BBCH 85 Aug 27	0	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	JSM0335 JSM0335-08

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg Parent	 NK-1375	 Tot.	Report; Trial no [ref]
2012 (H 342)					1 3 7	0.02/0.02 (0.02) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.03 (0.03) < 0.01 < 0.01	[Alé E., 2013m]
idem + [ADJ]	idem	40.7 41.8	10 10	idem	0 1 3 7	0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.02 < 0.01 < 0.01	idem
Pals, Spain, 2012 (Salis)	2 (10)	41.2 40.3	10 10	BBCH 65-89 Oct 01	1 3	0.02/0.01 (0.02) < 0.01/< 0.01 (< 0.01)[a]	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)[a]	0.03/ 0.02 (0.03) < 0.01/ < 0.01 (< 0.01)[a]	JSM0335 JSM0335-09 [Alé E., 2013m]
idem + [ADJ]	idem	42.2 41.2	10 10	idem	1 3	0.02 < 0.01	< 0.01 < 0.01	0.03 < 0.01	idem
Agia Marina, Greece, 2012 (Rio grande)	2 (11)	43.9 39.1	10 10	BBCH 86 Aug 07	0 1 3 7	0.01/0.01 (0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.02 < 0.01 < 0.01 < 0.01	JSM0335 JSM0335-10 [Alé E., 2013m]
idem + [ADJ]	idem	43.341.6	10 10	idem	0 1 3 7	0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01 < 0.01	idem
Fraz Rivalta Scrivia Tortona, Italy, 2013 (H 3406)	2 (11)	40.739.1	8 8	BBCH 88 Aug 16	0 1 3 8	0.02/0.02 (0.02) 0.02/0.01 (0.02) 0.01 < 0.01	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.03/ 0.03 (0.03) 0.03/ 0.02 (0.03) 0.02 < 0.01	JSM0486 JSM0486-07 [Alé E., 2013n]
Llambilles, Spain, 2013 (Ital rest)	2 (11)	37.9 39.5	8 8	BBCH 65-89 Aug 16	1 3	0.01/0.01 (0.01) < 0.01/< 0.01 (< 0.01)[b]	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)[b]	0.02/ 0.02 (0.02) < 0.01/ < 0.01 (< 0.01)	JSM0486 JSM0486-08 [Alé E., 2013n]

[ADJ] = commercial adjuvant codacide, oil seed rape oil 95%

[a] Plot size <30 m^3 (21.1 m^2)

[b] Plot size <30 m^3 (28 m^2)

A 50 g/L soluble concentrate (IKI-3106 50 SL aka IBE 4064) was applied on tomatoes in field residue trials in USA (21) and Canada (2) [Wiedmann & McDonald, report IB-2012-JLW-029-01-01]. Three spray applications of 60–97 g ai/ha with an interval of 6–8 days were applied at in a volume of 178–386 L/ha. An adjuvant was added to the mix at one test site (23). Trials were performed in 2012 and 2013 growing seasons and include two decline trials. Samples were picked at mature stage and included at least 12 fruits from 12 plants and/or weighed at least 2 kg, unless indicated differently [SS]. The results are summarised in Table 85.

Table 85 Supervised field trials tomatoes treated with a formulation with cyclaniliprole (50SL) using a pre-harvest backpack sprayer or tractor mounted boom sprayer

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg Parent	 NK-1375	 Tot.	Report; Trial no [ref]
North Rose, NY, USA,	3 (7)	61 60 61	20 20 20	green and red fruit	1	0.012/0.021 (0.016)	< 0.01/< 0.01 (< 0.01)	0.23/ 0.032 (0.030)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
2012 (Celebrity)				Sept 25					01-01-01 [Wiedmann &McDonald2014c]
Mebane, NC, USA, 2013 (Sun Gold Cherry)	3 (7)	81 81 81	29 29 29	BBCH 82 July 11	1	0.058/0.027 (0.043) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.069/ 0.038 (0.053) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-02 [Wiedmann &McDonald2014c]
Bradenton, FL, USA, 2013 (BHN 785)	3 (7)	88 87 84	47 47 47	fruit and flowers May 31	1	0.034/ 0.046 (0.040) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.045/ 0.057 (0.051) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-03 [Wiedmann &McDonald2014c]
Winter Garden, FL, USA, 2013 (Amelia)	3 (6-7)	80 80 81	21 21 21	BBCH 80-81 June 18	1	0.026/ 0.038 (0.032)	< 0.01/< 0.01 (< 0.01)	0.036/ 0.049 (0.042)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-04 [Wiedmann &McDonald2014c]
Northwood, ND, USA, 2013 (Better Boy)	3 (7)	80 79 79	28 34 28	BBCH 83 Sept 10	1	< 0.01/0.012 (0.011)	< 0.01/< 0.01 (< 0.01)	< 0.01/ 0.023 (0.022)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-07 [Wiedmann &McDonald2014c]
Verona, WI, USA, 2013 (Mountain Fresh)	3 (6-7)	81 80 81	30 31 31	BBCH 82-84 Sept 05	1	0.035/0.041 (0.038)	< 0.01/< 0.01 (< 0.01)	0.046/ 0.051 (0.049)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-08 [Wiedmann &McDonald2014c]
Seymour, IL, USA, 2013 (Celebrity)	3 (7-8)	80 79 80	29 29 28	BBCH 82 Sept 03	1	0.039/0.021 (0.030)	< 0.01/< 0.01 (< 0.01)	0.050/ 0.032 (0.041)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-09 [Wiedmann &McDonald2014c]
York, NE, USA, 2013 (Better Boy)	3 (7)	79 79 80	39 38 38	nr Sept 24	1	< 0.01/0.016 (0.013)	< 0.01/< 0.01 (< 0.01)	< 0.01/ 0.027 (0.024)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-10 [Wiedmann &McDonald2014c]
Pierron, IL, USA, 2013 (Jet Star)	3 (7)	61 61 60	22 22 23	BBCH 88 Sept 12	1	0.024/ 0.025 (0.024)	< 0.01/< 0.01 (< 0.01)	0.034/ 0.036 (0.035)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-11 [Wiedmann &McDonald2014c]
Wyoming, IL USA, 2013 (Delicious)	3 (7)	60 60 60	29 30 29	BBCH 83 Sept 10	1	0.021/0.028 (0.024)	0.024 / 0.028 (0.026)	0.031/ 0.039 (0.035)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-12 [Wiedmann &McDonald2014c]
Ladoga, IN, USA, 2013	3 (7-8)	79 79 79	38 38 38	some red tomatoes Aug 16	0 1	0.058/0.041 (0.049) [SS] 0.034/0.017/	< 0.01/< 0.01 (< 0.01) [SS] < 0.01/< 0.01/< 0.01/< 0.01	0.068/ 0.052 (0.060) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-13 and 14

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg Parent	NK-1375	Tot.	Report; Trial no [ref]
(Mountain Fresh)					3 7	0.036/0.022 (0.027)[a] [SS] 0.024/0.042 (0.033) [SS] 0.031/0.013 (0.022)	(< 0.01) [SS] < 0.01/0.015 (0.013) [SS] 0.014/< 0.01 (0.012)	0.045/ 0.027 (0.036) [SS] 0.034/ 0.058 (0.047) [SS] 0.045/ 0.024 (0.034)	[Wiedmann &McDonald2014c]
Geneva, MN, USA, 2013 (Super Fantastic)	3 (6-7)	79 81 81	39 39 42	fruiting Aug 30	1	0.038/ 0.030 (0.034)	< 0.01/< 0.01 (< 0.01)	0.048/ 0.041 (0.045)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-15 [Wiedmann &McDonald2014c]
Leonard, MO, USA, 2013 (Celebrity)	3 (7)	82 80 83	30 29 29	flower to ripe Aug 19	1	0.036/ 0.039 (0.037) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.046/ 0.050 (0.048) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-16 [Wiedmann &McDonald2014c]
Kerman, CA, USA, 2013 (Roma)	3 (7)	78 79 79	28 28 28	BBCH 75 Aug 19	1	0.070/ 0.083 (0.076) [SS]	0.024/0.028 (0.026) [SS]	0.095/ 0.11 (0.10) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-17 [Wiedmann &McDonald2014c]
Guadalupe, CA, USA, 2012 (Quait27)	3 (7)	60 60 60	23 23 20	BBCH 81 Oct 02	0 1 3 7	< 0.01/0.014 (0.012) 0.025/0.011 (0.018) 0.016/0.017 (0.017) [SS] 0.016/< 0.01 (0.013)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) [SS] < 0.01/< 0.01 (< 0.01)	< 0.01/ 0.025 (0.023) 0.035/ 0.022 (0.029) 0.026/ 0.028 (0.027) [SS] 0.027/ < 0.01 (0.024)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-18 [Wiedmann &McDonald2014c]
Guadalupe, CA, USA, 2013 (RomaK2763)	3 (7)	79 80 80	27 27 27	BBCH 84 Oct 29	1	0.036/0.029 (0.032) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.046/ 0.040 (0.043) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-19 [Wiedmann &McDonald2014c]
Porterville, CA, USA, 2013 (AB2)	3 (6-7)	80 80 79	27 25 25	BBCH 89 Aug 26	1	0.026/0.025 (0.026)	< 0.01/< 0.01 (< 0.01)	0.036/ 0.036 (0.036)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-20 [Wiedmann &McDonald2014c]
Sanger, CA, USA, 2013 (Q21)	3 (7)	62 97 83	16 27 22	BBCH 87 Sept 13	1	0.044/0.041 (0.042)	< 0.01/< 0.01 (< 0.01)	0.055/ 0.051 (0.053)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-21 [Wiedmann

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
									&McDonald2014c]
Dunnigan, CA, USA, 2013 (577 Heinz)	3 (7)	81 81 81	43 43 43	BBCH 89 Sept 01	1	0.025/0.028 (0.026)	< 0.01/< 0.01 (< 0.01)	0.035/ 0.039 (0.037)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-22 [Wiedmann &McDonald2014c]
Madera, CA, USA, 2013 (Quality) + [ADJ]	3 (7)	81 81 81	29 29 29	Mature tomatoes July 15	1	0.027/0.031 (0.029)	< 0.01/0.011 (0.011)	0.038/ 0.043 (0.040)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-23 [Wiedmann &McDonald2014c]
Branchton, Ontario, CANADA, 2013 (TSH24)	3 (6-7)	81 80 79	40 40 40	BBCH 85 Aug 29	1	0.063/0.076 (0.070) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.074/ 0.087 (0.080) [SS]	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-05 [Wiedmann &McDonald2014c]
St-Marc-sur-Richelieu, Quebec, CANADA, 2013 (Joker)	3 (6-8)	81 80 73	32 32 32	BBCH 73 Aug 22	1	0.021/0.016 (0.019)	< 0.01/< 0.01 (< 0.01)	0.032/ 0.027 (0.029)	IB-2012-JLW-029-01-01 IB-2012-JLW-029-01-01-06 [Wiedmann &McDonald2014c]

[ADJ] = Adjuvant (R-11 Adjuvant (site 23)) was added with application.

[SS] sample size below the required 2 kg), but >1.25 kg, with at least 12 fruits picked from at least 12 plants/locations on the plot. Samples were considered suitable enough to be used for maximum residue estimation.

[a] Mean of four replicate field samples

A 50 g/L soluble concentrate of cyclaniliprole (4.5%) was applied on cherry tomatoes in field residue trials in Japan (six decline trials) [Kouji, N, 2012, report JP2011C132, Yoshiyuki T, 2013a, report JP2012C105, and Yoshiyuki T, 2013b, report JP2012C106]. Two foliar spray applications of 111–141 g ai/ha were applied at a volume of 2220–2810 L/ha (222–281 L/10 are) with a back pack sprayer. Trials were performed in the 2011 and 2012 growing seasons; sampling days ranged from 1–21 days. Tomatoes were harvested at late harvesting phase and samples consisted of 10–14 pieces, and/or 1.3–3.4 kg. The results are summarised in Table 86.

Table 86 Supervised field trials on tomato treated with two pre-harvest foliar spray formulation with cyclaniliprole (50SL)

TOMATO Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Motomiya, Morioka-shi, Iwate, Japan, 2011 (Saturn)	2 (7)	111	5	fruit growing phase, July 26	1	0.05 / 0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.06/ 0.06 (0.06)	Iwate JP2011C132A [Kouji N, 2012]
					3	0.04 / 0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.05)	

TOMATO Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
					7	0.03 / 0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	
					14	0.04 / 0.03 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.04 (0.05)	
					21	0.02 / 0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	
Hiragi-machi, Hakusan-shi, Ishikawa, Japam, 2011 (Momotaro haruka)	2 (7)	125	5	initial harvesting phase, June 2	1	0.05 / 0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.06/ 0.06 (0.06)	Ishikawa JP2011C132B [Kouji N, 2012] [Koki M, 2012]
					3	0.05 / 0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.06/ 0.06 (0.06)	
					7	0.04 / 0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.05)	
					14	0.04 / 0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.05)	
					21	0.03 / 0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	
Nukazuka, Hanamaki-shi, Iwate, Japan, 2012 (Carol 10 (cherry tomato))	2 (7)	111	5	growing phase, July 23	1	0.25 / 0.24 (0.25) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.26/ 0.025 (0.026) [SS]	Iwate JP2012C105A [Yoshiyuki, 2013a, report JP2012C105]
					3	0.25 / 0.24 (0.25) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.26/ 0.25 (0.26) [SS]	
					7	0.15 / 0.15 (0.15) [SS]	0.02 / 0.02 (0.02) [SS]	0.17/ 0.17 (0.17) [SS]	
					14	0.09 / 0.09 (0.09) [SS]	0.02 / 0.02 (0.02) [SS]	0.11/ 0.11 (0.11) [SS]	
					21	0.05 / 0.05 (0.05) [SS]	0.01 / 0.01 (0.01) [SS]	0.06/ 0.06 (0.06) [SS]	
Yokote-machi, Maebashi-shi, Gunma, Japan, 2012 (Suncherry pure (cherry tomato))	2 (7)	125	5	Late harvesting phase, June 11	1	0.14 /0.14 (0.14) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.15/ 0.15 (0.15) [SS]	Gunma JP2012C105B [Yoshiyuki, 2013a, report JP2012C105]
					3	0.13 / 0.13 (0.13) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.14/ 0.14 (0.14) [SS]	
					7	0.11 / 0.11 (0.11) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.12 / 0.12 (0.12) [SS]	
					14	0.13 / 0.12 (0.13) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.14 / 0.13 (0.14) [SS]	
					21	0.09 / 0.09 (0.09) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.10 / 0.10 (0.10) [SS]	

TOMATO Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
535 Kessoku-cho, Ushiku-shi, Ibaraki, Japan, 2012 (Pepe (cherry tomato))	2 (7)	141	5	harvesting phase, Jan 14	1	0.25 / 0.23 (0.24) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.26 / 0.24 (0.25) [SS]	Ibaraki JP2012C105C [Yoshiyuki, 2013a, report JP2012C105]
					3	0.29 / 0.29 (0.29) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.30 / 0.30 (0.30) [SS]	
					7	0.13 / 0.13 (0.13) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.14 / 0.14 (0.14) [SS]	
					14	0.18 / 0.18 (0.18) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.19 / 0.19 (0.19) [SS]	
					21	0.19 / 0.18 (0.19) [SS]	< 0.01/< 0.01 (< 0.01) [SS]	0.20 / 0.19 (0.20) [SS]	
Ikenouchi, Sakurai-shi, Nara, Japan, 2012 (Saturn)	2 (7)	139	5	harvesting phase, June 26	1	0.08 / 0.07 (0.08)	< 0.01/< 0.01 (< 0.01)	0.09/ 0.08 (0.09)	Nara JP2012C106A [Yoshiyuki, 2013b, report JP2012C106]
					3	0.07 / 0.07 (0.07)	< 0.01/< 0.01 (< 0.01)	0.08/ 0.08 (0.08)	
					7	0.06 / 0.06 (0.06)	< 0.01/< 0.01 (< 0.01)	0.07/ 0.07 (0.07)	
					14	0.05 / 0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.06/ 0.06 (0.06)	
					21	0.04 / 0.04 (0.04)	0.01 / 0.01 (0.01)	0.05/ 0.05 (0.05)	

[SS] = sample size below the required 2 kg (1.2-1.9 kg)

Tomato, indoor applications

A 50 g/L soluble concentrate (IKI-3106 50 SL aka IBE 4064) was applied in eight European residue indoor trials on tomatoes [Alé, 2013o, report JSM0353, Alé, 2013p, report JSM0354, Alé, 2013q, report JSM0488]. Two groundboom or lance spray applications (38.9–44.4 g ai/ha) with an interval of 6–8 days were applied at a volume of 954–1090 L/ha. Trials were performed in North and South Europe and include four decline trials. Report JSM00353 [Alé, 2013m] was a comparative study, where on one plot of each test site, in addition to the regular treatment, an adjuvant [ADJ] was applied. The results are summarised in Table 87.

Table 87 Supervised indoor greenhouse trials on tomatoes treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL) with or without adjuvant

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Belfeld, the Netherlands, 2012 (Capricia RZ)	2 (6)	41.6 38.9	4 4	BBCH 87-89 Oct 16	0 1 3 7	0.06/0.06 (0.06) 0.05/0.05 (0.05) 0.04 0.03	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.07/ 0.07 (0.07) 0.06/ 0.06 (0.06) 0.05 0.04	JSM0353 JSM0353-01 [Alé E., 2013o]
idem + [ADJ]	idem	40.2 42.6	4 4	idem	0 1 3 7	0.05 0.04 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01	0.06 0.05 0.04 0.05	idem
Bouloc, France, 2012 (Coeur de boeuf)	2 (7)	43.1 43.9	4 4	BBCH 89 Sept 11	1 3	0.02/0.02 (0.02) 0.02/0.02 (0.02)[a]	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)[a]	0.03/ 0.03 (0.03) 0.03/ 0.03 (0.03)[a]	JSM0353 JSM0353-02 [Alé E., 2013o]
idem + [ADJ]	idem	42.7 43.1	4 4	idem	1 3	0.03 0.04	< 0.01 < 0.01	0.04 0.05	idem
Wellerllooi, the Netherlands, 2012 (270-202RZ)	2 (7)	40.739 .8	4 4	BBCH 87-89 Sept 10	0 1 3 7	0.05/0.05 (0.05) 0.05/0.05 (0.05) 0.04 0.04	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.06/ 0.06 (0.06) 0.06/ 0.06 (0.06) 0.05 0.05	JSM0354 JSM0354-01 [Alé E., 2013p]
idem + [ADJ]	idem	40.439 .3	4 4	idem	0 1 3 7	0.04 0.04 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.05 0.04 0.05	idem
Fronton, France, 2012 (Corazon)	2 (7)	43.9 43.9	4 4	BBCH 89 Sept 11	1 3	< 0.01/< 0.01 (< 0.01) < 0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01) < 0.01/ 0.02 (0.02)	JSM0354 JSM0354-02 [Alé E., 2013p]
idem + [ADJ]	idem	43.542 .3	4 4	idem	1 3	0.02 0.05	< 0.01 < 0.01	0.03 0.06	idem
Baldisero D'Álba, Italy, 2012 (Rugantino)	2 (6)	40.9 40.9	4 4	BBCH 83 Sept 11	1 3	0.03/0.03 (0.03) 0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04) 0.03/ 0.03 (0.03)	JSM0354 JSM0354-03 [Alé E., 2013p]
idem + [ADJ]	idem	41.0 44.4	4 4	idem	1 3	0.04 0.03	< 0.01 < 0.01	0.05 0.04	idem
Bra, Italy, 2012 (Ingrid)	2 (7)	40.7 40.9	4 4	BBCH 89 Oct 29	0 1 3 7	0.01/0.03 (0.02) 0.03/0.02 (0.03) 0.01 0.02	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.02/ 0.04 (0.03) 0.04/ 0.03 (0.04) 0.02 0.02	JSM0354 JSM0354-04 [Alé E., 2013p]
idem + [ADJ]	idem	40.5 40.7	4 4	idem	0 1 3 7	0.02 0.02 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.03 0.02 0.03	idem

TOMATO Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Wellerlooi, the Netherlands, 2013 (Capricia)	2 (8)	40.9 40.2	4 4	BBCH 87-89 June 28	0 1 3 8	0.04/0.04 (0.04) 0.04/0.04 (0.04) 0.05 0.05	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01) < 0.01 < 0.01	0.05/ 0.05 (0.05) 0.05/ 0.05 (0.05) 0.06 0.06	JSM0488 JSM0488-01 [Alé E., 2013q]
Barranquete, Spain, 2013 (Ramile)	2 (8)	42.6 41.2	4 4	BBCH 85 March 31	1 3	0.04/0.03 (0.04) 0.03/0.03 (0.03)[b]	< 0.01/< 0.01 (< 0.01) < 0.01/< 0.01 (< 0.01)[b]	0.05/ 0.04 (0.05) 0.04/ 0.04 (0.04)[b]	JSM0488 JSM0488-02 [Alé E., 2013q]

[ADJ] = commercial adjuvant codacide, oil seed rape oil 95%

[a] Plot size <30 m^3 (20.4 m^2)

[b] plot size <30 m^3 (20 m^2)

Leafy vegetables (including Brassica leafy vegetables)

Lettuce (head & leafy)

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on head lettuce and leaf lettuce in field residue trials in USA (8/8) and Canada (2/2) [McDonald & Wiedmann, 2014c, report IB-2013-JAM-001-01-01]. Three spray applications of 61–100 g ai/ha with an interval of 6–8 days were applied at in a volume of 180–390 L/ha. At two sites an adjuvant was added to the mix. Trials were performed in 2012 and 2013 growing seasons and include one decline trial in leaf lettuce. Samples were picked at mature stage (BBCH 48–55), except for two trials on leafy lettuce (BBCH 19+). The results on head lettuce (with wrapper leaves and without wrapper leaves) and leafy lettuce are presented in Table 88 and Table 89, respectively.

Table 88 Supervised field trials on lettuce (head) treated with a formulation with cyclaniliprole (50SL) using a pre-harvest back pack or custom-made sprayer. Residues are presented for head wwl or w/owl (with or without wrapper leaves)

LETTUCE, HEAD Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
North Rose, NY, USA, 2013 (Ithaca) [WWL]	3 (7,7)	83 81 83	33 32 33	10-15 cm heads, July 25	1	1.1/1.2 (1.1)	0.19/0.20 (0.19)	1.3/ 1.4 (1.3)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-01 [McDonald& Wiedmann, 2014b]
idem [W/OWL]						0.60/0.61 (0.60)	0.10/0.11 (0.11)	0.71/ 0.73 (0.72)	idem
Bradenton, FL, USA, 2012 (Raider) [WWL]	3 (7)	68 67 68	24 24 24	head formation Dec 16	2	0.37/0.27 (0.32)	0.040/0.037 (0.039)	0.41/ 0.31 (0.36)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-03 [McDonald&Wiedmann, 2014b]
idem [W/OWL]						0.065/0.11 (0.086)	< 0.01/0.014 (0.012)	0.075/ 0.12 (0.098)	idem

LETTUCE, HEAD Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Seymour, IL, USA, 2013 (Paris Ilands cos) [WWL]	3 (7)	83 86 81	28 28 29	BBCH 60 Aug 08	1	0.77/1.1 (0.92)	0.096/0.12 (0.11)	0.87/ 1.2 (1.0)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-05 [McDonald&Wiedmann, 2014b]
idem [W/OWL]						0.72/0.69 (0.71)	0.045/0.033 (0.039)	0.77/ 0.73 (0.75)	idem
Guadalupe, CA, USA, 2013 (Iceberg) [WWL]	3 (6,7)	82 78 82	28 28 28	BBCH 49 July 12	1	1.2/1.5 (1.4)	0.21/0.27 (0.24)	1.5/ 1.8 (1.6)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-15 [McDonald&Wiedmann, 2014b]
idem [W/OWL]						0.081/0.097 (0.089)	0.013/0.016 (0.015)	0.095/ 0.11 (0.10)	idem
Porterville, CA, USA, 2013 (Vandenberg) [WWL]	3 (6,8)	81 80 80	29 29 29	BBCH 49 May 13	1	0.20/0.32 (0.26)	0.034/0.051 (0.043)	0.24/ 0.38 (0.31)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-17 [McDonald&Wiedmann, 2014b]
idem [W/OWL]						0.012/< 0.01 (0.011)	< 0.01/< 0.01 (< 0.01)	0.023/ 0.021 (0.022)	idem
Sanger, CA, USA, 2012 (Tahema) [WWL]	3 (7,7)	61 62 63	16 16 16	BBCH 49 Nov 02	1	0.55/0.57 (0.56)	0.045/0.050 (0.048)	0.60/ 0.62 (0.61)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-19 [McDonald&Wiedmann, 2014b]
idem [W/OWL]						0.059/0.063 (0.061)	< 0.01/< 0.01 (< 0.01)	0.070/ 0.074 (0.072)	idem
Madera, CA, USA, 2013 (Great lakes) [WWL] + [ADJ]	3 (7,7)	62 62 61	22 22 21	mature head lettuce Jan 16	1	2.0/2.3 (2.2)	0.14/0.14 (0.14)	2.2/ 2.4 (2.3)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-21 [McDonald&Wiedmann, 2014b]
idem [W/OWL]					1	0.16/0.10 (0.13)	< 0.01/< 0.01 (< 0.01)	0.18/ 0.11 (0.14)	idem
Princeton, Ontario, CANADA, 2013 (Butterhead) [WWL]	3 (7,7)	74 76 86	32 32 32	BBCH 47-49 July 31	1	0.097/0.090 (0.094)	0.013/0.011 (0.012)	0.11/ 0.10 (0.11)	IB-2012-JAM-001-01-01 IB-2012- JAM-00-01-01-09 [McDonald&Wiedmann, 2014b]
idem					1	0.037/0.042	< 0.01/< 0.01	0.048/ 0.053	idem

LETTUCE, HEAD Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
[W/OWL]						(0.040)	(< 0.01)	(0.050)	
St-Marc-sur-Richelieu, Quebec, CANADA, 2013 (PYB 7101) [WWL]	3 (7,8)	79 78 78	40 40 40	BBCH 48, July 18	1	0.065/0.069 (0.067)	0.024/0.030 (0.027)	0.091/ 0.10 (0.096)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-11 [McDonald&Wiedmann, 2014b]
idem [W/OWL]					1	< 0.01/ < 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem

[ADJ] = Adjuvant (IR-11 Adjuvant (site 21) was added with application.

Table 89 Supervised field trials on lettuce (leafy) treated with a formulation with cyclaniliprole (50SL) using a pre-harvest back pack or custom-made sprayer.

LETTUCE, LEAFY Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Seven Springs, NC, USA, 2012 (Black Seeded Simpson)	3 (6,7)	80 80 78	3632 31	BBCH 49 May 08	1	2.7/3.2 (3.0)	0.27/0.31 (0.29)	3.0/ 3.6 (3.3)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-02 [McDonald&Wiedman n, 2014b]
Bradenton, RL, USA, 2012 (Green Tower, Romaine)	3 (7,7)	67 66 66	24 24 24	Heading/ flowering initiation Dec 16	1	0.71 / 0.76 (0.74)	0.10 / 0.11 (0.10)	0.82/ 0.87 (0.85)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-04 [McDonald&Wiedman n, 2014b]
Seymour, IL, USA, 2013 (Black Seeded Simpson)	3 (6,9)	79 80 78	29 29 29	BBCH 49 July 23	1	2.5 / 2.4 (2.4)	0.18 / 0.19 (0.19)	2.7/ 2.6 (2.6)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-06 [McDonald&Wiedman n, 2014b]
Highland, IL, USA, 2013 (Oakleaf)	3 (6, 7)	81 81 81	29 29 29	BBCH 49 Nov 04	1	0.77 / 0.77 (0.77)	0.022/ 0.022 (0.022)	0.80/ 0.79 (0.79)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-08 [McDonald&Wiedman n, 2014b]
Northwood, ND, USA, 2013 (Romaine)	3 (7,7)	81 79 80	32 32 32	BBCH 19+	1	0.83/0.70 (0.76)	0.084/0.070 (0.077)	0.92/ 0.77 (0.84)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-14 [McDonald&Wiedman n, 2014b]

LETTUCE, LEAFY Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Glenn, CA, IUSA, 2012 (Greenleaf)	3 (6,8)	61 61 61	32 32 32	BBCH 49 Nov 27	1	1.48/1.1 (1.2)	0.037/0.028 (0.033)	1.4/ 1.1 (1.3)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-16 [McDonald&Wiedmann, 2014b]
Porterville, CA, USA, 2013 (Star fighter)	3 (7,7)	81 80 80	29 29 29	BBCH 49 Feb 28	1	1.3/1.2 (1.3)	0.086/ 0.074 (0.080)	1.4/ 1.3 (1.4)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-18 [McDonald&Wiedmann, 2014b]
Sanger CA, USA, 2013 (Green Forest)	3 (7,7)	63 100 83	16 26 22	BBCH 48 June 03	1	0.91/0.81 (0.86)	0.16 / 0.16 (0.16)	1.1/ 0.98 (1.0)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-20 [McDonald&Wiedmann, 2014b]
Madera, CA, USA, 2012 (Green Star) + [ADJ]	3 (7,7)	61 61 61	22 22 21	mature lettuce, Nov 01	1	1.9/2.1 (2.0)	0.19 / 0.21 (0.20)	2.1/ 2.3 (2.2)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-22 [McDonald&Wiedmann, 2014b]
Princeton, Ontario, CANADA, 2013 (Butterhead)	3 (6,7)	79 82 84	32 32 32	BBCH 49 June 25	0 1 3 7	1.2 / 1.3 (1.2) 0.26 / 0.23 (0.25) 0.088/0.095 (0.092) 0.031/0.031 (0.031)	0.027/0.028 (0.028) 0.024/0.020 (0.022) 0.012/0.010 (0.011) < 0.01/< 0.01 (< 0.01)	1.2/ 1.3 (1.3) 0.28/ 0.25 (0.27) 0.10/ 0.11 (0.10) 0.041 / 0.041 (0.41)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-10 [McDonald&Wiedmann, 2014b]
St-Marc-sur-Richelieu, QC CANADA 2013 (Bronco)	3 (7,8)	80 71 85	40 40 40	BBCH 19 July 03	1	2.1 / 2.3 (2.2)	0.35/0.36 (0.36)	2.5/ 2.6 (2.6)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-12 [McDonald&Wiedmann, 2014b]

[ADJ] = Adjuvant (IR-11 Adjuvant (site 22)) was added with application.

[GS] growth stage not representative for commercial harvest.

Spinach

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on spinach in field residue trials in USA (8) and Canada (1) [McDonald & Wiedmann, 2014c, report IB-2013-

JAM-001-01-01]. Three spray applications of 60–83 g ai/ha with an interval of 6–8 days were applied at in a volume of 178–319 L/ha. At one site an adjuvant was added to the mix. Trials were performed in 2012 and 2013 growing seasons and did not include a decline trial. Sample sizes were collected from at least 12 different areas and weighed 1.0–1.81 kg, except the sample from Canada with 0.5 kg. Samples were picked at mature stage (BBCH 48–55), except the trial from Canada (BBCH 16–19). The results are summarised in Table 90.

Table 90 Supervised field trials on spinach treated with a formulation with cyclaniliprole (50SL) using a pre-harvest back pack or custom-made sprayer

SPINACH Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Germansville, PA, USA, 2013 (Tyee)	3 (6,8)	82 80 80	39 39 39	BBCH 48-49 June 04	1	3.2/3.6 (3.4)	0.68/0.70 (0.69)	4.0/ 4.3 (4.1)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-23 [McDonald&Wiedmann, 2014b]
Seven Springs, NC, USA, 2012 (Baker)	3 (6,8)	61 60 60	21 20 20	BBCH 49 Oct 31	1	3.0/2.8 (2.9)	0.36/0.32 (0.34)	3.3/ 3.2 (3.3)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-24 [McDonald&Wiedmann, 2014b]
Dearfield, MI, USA, 2013 (Crocodile Hybrid)	3 (7,7)	61 60 61	26 26 26	nr Oct 23	2	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/ < 0.01 (< 0.01)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-26 [McDonald&Wiedmann, 2014b]
Seymour, IL, USA, 2013 (Olympia F1)	3 (7,7)	82 83 80	29 29 29	BBCH 55 Aug 22	1	2.8/2.7 (2.8)	0.50/0.58 (0.54)	3.3/ 3.3 (3.3)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-27 [McDonald&Wiedmann, 2014b]
Uvalde, TX, USA, 2012 (DMC 6607)	3 (7,7)	62 61 62	32 33 32	BBCH 48 Dec 19	1	2.2/2.3 (2.2)	0.20/0.21 (0.21)	2.4/ 2.5 (2.4)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-28 [McDonald&Wiedmann, 2014b]
Jerome, ID, USA, 2012 (Unipack 151)	3 (6,8)	63 62 63	21 20 20	BBCH 49 Oct 24	1	1.9/2.1 (2.0)	0.068/0.072 (0.070)	1.9/ 2.1 (2.0)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-29 [McDonald&Wiedmann, 2014b]
Santa Maria, USA, 2013 (Avenger) + [ADJ]	3 (6,7)	80 80 82	28 28 28	BBCH 49 July 12	1	2.4/2.4 (2.4)	0.25/0.24 (0.25)	2.6/ 2.6 (2.6)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-30 [McDonald&Wiedmann, 2014b]
Madera, CA, USA, 2012	3 (7,7)	61 61 61	22 22 21	mature spinach Dec 27	1	1.4/1.4 (1.4)	0.079/0.068 (0.074)	1.5/ 1.5 (1.5)	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-31

SPINACH Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
(Shasta) + [ADJ]									[McDonald&Wiedmann, 2014b]
St-Marc-sur-Richelieu, QC CANADA 2013 (Bronco)	3 (7,8)	80 71 85	40 40 40	BBCH 16-19 July 03	1	4.5/4.7 (4.6) [SS][GS]	0.88/0.89 (0.88) [SS][GS]	5.4/ 5.7 (5.5) [SS][GS]	IB-2012-JAM-001-01-01 IB-2012-JAM-00-01-01-25 [McDonald&Wiedmann, 2014b]

[ADJ] = Adjuvant (IR-11 Adjuvant) was added with application.

[SS] = sample size 0.5 kg instead of required 1 kg.

[GS] = growth stage not representative for commercial harvest

Chinese cabbage

A 50 g/L soluble concentrate of cyclaniliprole (4.5%) was applied on Chinese cabbage in field residue trials in Japan (six decline trials) [Yoshiyuki T, 2013c, report JP2012C108, and Hitoshi I, 2013, report JP2013C091]. Two foliar spray applications of 50–73 g ai/ha were applied at a volume of 2000–2930 L/ha (200–290 L/10 are) with a backpack sprayer. Trials were performed in the 2012 and 2013 growing seasons; sampling days ranged from 1–7 and 14–21days on two different plots at the same location. Cabbages were harvested at different growth stages and samples consisted of 5–8 pieces, weighing 5.7–17.5 kg. The results are summarised in Table 91.

Table 91 Supervised field trials on Chinese cabbage treated with two pre-harvest foliar spray formulations with cyclaniliprole (50SL) by back pack spraying

CHINESE CABBAGE Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
82-9 Tanaka, Kuroishi-shi, Aomori, Japan, 2012 (Shunju)	2 (7)	50 50	3 3	growth of head Oct 01	loam	1	0.37 / 0.34 (0.36)	< 0.01/< 0.01 (< 0.01)	0.38/ 0.35 (0.37)	Aomori JP2012C108A [Yoshiyuki, 2013c, report JP2012C108]
						3	0.15 / 0.14 (0.15)	< 0.01/< 0.01 (< 0.01)	0.16/ 0.15 (0.16)	
						7	0.11 / 0.11 (0.11)	< 0.01/< 0.01 (< 0.01)	0.12/ 0.12 (0.12)	
idem	idem	50 50	3 3	beginning of head forming Sept 17	idem	14	0.04 / 0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.5)	idem
						21	0.01 / 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	
Kihisho, Echizencho, Nyu-gun, Fukui, Japan, 2012 (Kigokoro)	2 (7)	67 67	3 3	middle of head forming Oct. 17	sandy loam	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	Fukui JP2012C108B [Yoshiyuki, 2013c, report JP2012C108]
						3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
						7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	

CHINESE CABBAGE Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
idem	idem	60 60	3 3	growing season Oct 03		14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
						21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
535 Kessoku-cho, Ushiku-shi, Ibaraki Japan, 2013 (Kiraboshi 65)	2 (7)	63 63	2 2	harvesting period June 10	loam	1	0.15 / 0.14 (0.15)	< 0.01/< 0.01 (< 0.01)	0.16/ 0.15 (0.16)	Ibaraki JP2013C091A [Hitoshi, 2013, report JP2013C091]
						3	0.02 / 0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	
						7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (6)	56 63	3 3	head-forming stage March 27		14	0.01/ < 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/< 0.01 (0.02)	idem
						21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
2476 Shimoichida, Takamori-machi, Shimoina-gun, Nagano, Japan, 2013 (Harusakari)	2 (7)	73 73	2 2	ripening stage June 03	sandy loam	1	0.17 / 0.17 (0.17)	0.01/0.01 (0.01)	0.18/ 0.18 (0.18)	Nanshin JP2013C091B [Hitoshi, 2013, report JP2013C091]
						3	0.13 / 0.13 (0.13)	0.02/0.02 (0.02)	0.15/ 0.15 (0.15)	
						7	0.06 / 0.06 (0.06)	0.02/0.01 (0.02)	0.08/ 0.07 (0.08)	
idem	2 (7)	72 72	2 2	mid head forming March 20		14	0.01 / 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	idem
						21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
Ureshinoshimada-cho, Matsusaka-shi, Mie, Japan, 2013 (Daifuku)	2 (8)	63 63	3 3	head forming stage Oct 18	loam	1	0.05 / 0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.06/ 0.06 (0.06)	Mie JP2013C091C [Hitoshi, 2013, report JP2013C091]
						3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
						7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (6)	63 63	3 3	head forming stage Oct 03	idem	14	0.02 / 0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	idem
						21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	

CHINESE CABBAGE Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
Nukazuka, Hanamaki-shi, Iwate, Japan, 2013 (Musohak-usai)	2 (7)	57 57	3 3	growing season Oct 29	clay loam	1	0.11 / 0.10 (0.11)	< 0.01/< 0.01 (< 0.01)	0.12/ 0.11 (0.12)	Iwate JP2013C091E [Hitoshi, 2013, report JP2013C091]
						3	0.04 / 0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05/ 0.05 (0.05)	
						7	0.02 / 0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	
idem	2 (7)	57 57	3 3	growing season Oct 15		14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
						21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	

Kale

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied in 4 European residue trials on kale [Alé, 2013r, report JSM0483]. Two ground directed boomspray applications (24.5–27.7 g ai/ha) with an interval of 13–14 days were applied at in a volume of 268–313 L/ha. Trials were performed in Northern Europe and include two decline trials. Single or duplicate field samples consisted of at least 12 plants and weighed >2 kg from PHI 13 and longer. The results are summarised in Table 92.

Table 92 Supervised field trials on kale (leaves with stems) treated with a pre-harvest foliar spray formulation with cyclaniliprole (50SL)

KALE Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS&last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Goch-Nierswalde, Germany, 2013 (Reflex)	2 (14)	25.825.6	8 8	BBCH 47 Aug 23	0 3 7 14 21	0.56/ 0.41 (0.49)[SS] 0.22 [SS] 0.23 [SS] 0.13/ 0.14 (0.14) 0.02	0.02/ 0.01 (0.02) [SS] 0.03 [SS] 0.04 [SS] 0.03 / 0.03 (0.03) < 0.01	0.58/ 0.42 (0.51)[SS] 0.25 [SS] 0.27 [SS] 0.16/ 0.17 (0.17) 0.03	JSM0483 JSM0483-01 [Alé E, 2013r]
Wunstorf, Germany, 2013 (Westländer halbhoher)	2 (14)	27.3 27.7	10 10	BBCH 46-47 Sept 05	14	0.01/ 0.02 (0.02)	< 0.01/ < 0.01 (< 0.01)	0.02/ 0.03 (0.03)	JSM0483 JSM0483-02 [Alé E, 2013r]

KALE Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS&last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Ottersum, the Netherlands, 2013 (Reflex)	2 (14)	24.6 24.5	8 8	BBCH 47 Aug 23	0 3 7 13 21	0.38/ 0.44 (0.41) [SS] 0.22 [SS] 0.21 [SS] 0.20/ 0.14 (0.17) 0.03	0.01/ 0.01 (0.01) [SS] 0.02 [SS] 0.03 [SS] 0.04 /0.03 (0.04) < 0.01	0.39/ 0.45 (0.42) [SS] 0.24 [SS] 0.24 [SS] 0.24/ 0.17 (0.21) 0.04	JSM0483 JSM0483-03 [Alé E, 2013r]
Grubbenvorst, the Netherlands, 2013 (Ripbor)	2 (13)	26.526.5	8 8	BBCH 47 Sept 27	13	0.05/ 0.06 (0.06)	0.01 /0.01 (0.01)	0.061/ 0.071 (0.66)	JSM0483 JSM0483-04 [Alé E, 2013r]

[SS] sample size below the required 2 kg.

Mustard greens

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on mustard greens in field residue trials in USA (5) [Wiedmann & McDonald, report IB-2013-JLW-028-01-01]. Three spray applications of 60–81 g ai/ha with an interval of 6–7 days were applied at in a volume of 205–322 L/ha. An adjuvant was added to the mix at one test site. Trials were performed in 2012 (3) and 2013 (2) growing seasons and include one decline trial. The results are summarised in Table 93.

Table 93 Supervised field trials on mustard greens treated with a formulation with cyclaniliprole (50SL) using a pre-harvest backpack sprayer, homemade sprayer or tractor mounted boom sprayer

MUSTARD GREENS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
Seven Springs, USA, 2012 (Tendergreen) + [ADJ]	3 (6,7)	60 61 60	21 21 20	BBCH 49 Oct 23	loamy sand	0 1 3 7	4.8/5.1 (5.0) 3.1/3.0 (3.0) 2.1/2.4 (2.3) 0.80/0.82 (0.81)	0.39/0.39 (0.39) 0.42/0.40 (0.41) 0.40/0.45 (0.43) 0.16/0.17 (0.16)	5.2/ 5.5 (5.4) 3.5/ 3.4 (3.5) 2.5/ 2.9 (2.7) 0.97/ 1.0 (0.99)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-21 [Wiedmann &McDonald2014b]
Cheneyville, LA, USA, 2012 (Florida Broadleaf)	3 (7,7)	62 61 60	20 20 19	8-10 leaf Nov 13	silt	1	4.0/4.2 (4.1)	0.30/0.32 (0.31)	4.3/ 4.6 (4.4)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-22 [Wiedmann &McDonald2014b]

MUSTARD GREENS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
Leonard, MO, USA, 2013 (Southern Giant Curled)	3 (7,7)	81 80 79	39 39 39	some plants heading June 27	silty clay loam	1	4.1/3.8 (4.0)	0.36/0.32 (0.34)	4.5/ 4.1 (4.3)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-23 [Wiedmann &McDonald2014b]
Hinton, OK, USA, 2013 (Florida Broadleaf)	3 (6,7)	80 80 80	38 38 38	BBCH 45 Nov20	sandy loam	1	5.3/6.5 (5.9)	0.30/0.35 (0.32)	5.6/ 6.8 (6.2)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-24 [Wiedmann &McDonald2014b]
Madera, CA, USA, 2013 (Florida Broadleaf) + [ADJ]	3 (7,7)	61 61 61	22 22 21	mature mustard greens Nov 01	loamy sand	1	1.3/1.5 (1.4)	0.095/0.10 (0.10)	1.4/ 1.6 (1.5)	IB-2012-JLW-028-01-01 IB-2012-JLW-028-01-01-25 [Wiedmann &McDonald2014b]

[ADJ] = Adjuvant (R-11 Adjuvant (site 25)) was added with application.

Soya bean, immature (with pods)

A 50 g/L soluble concentrate of cyclaniliprole (4.5%) was applied on soya bean in field residue trials in Japan (three decline trials) [Takashi N, 2012a, report JP2011C362 and Yoshiyuki T, 2013d, report JP2012C103]. With an interval of 7 days two foliar spray applications of 38–50 g ai/ha were applied at a volume of 1500–2000 L/ha with a back pack sprayer. Trials were performed in the 2011 and 2012 growing seasons; sampling days ranged from 1–21 days. The samples were air dried in a greenhouse for 5–8 days, followed by threshing and subsequent sorting by wind in a winnowing basket. The results are summarised in Table 94.

Table 94 Supervised field trials on soya bean (green/immature seeds) treated with two pre-harvest foliar spray formulations with cyclaniliprole (50SL) by back pack spraying

SOYA BEAN, GREEN Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
82-9 Tanaka, Kuroishi-shi, Aomori, Japan, 2011 (Aomori Toyomaru)	2 (7)	38 38	3 3	grain enlargement stage Aug 27	1	0.10 / 0.10 (0.10)	< 0.01/< 0.01 (< 0.01)	0.11/ 0.11 (0.11)	Aomori JP2011C362A [Takashi, 2012a, report JP2011C362]
					3	0.07 / 0.06 (0.06)	< 0.01/< 0.01 (< 0.01)	0.08/ 0.07 (0.07)	
					7	0.04 / 0.04	< 0.01/< 0.01	0.05/ 0.05	

SOYA BEAN, GREEN Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
						(0.04)	(< 0.01)	(0.05)	
idem	2 (7)	38 38	3 3	grain enlargement stage Aug 14	14	0.01 / 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	idem
					21	0.01 / 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/ 0.02 (0.02)	
Tsubone, Murakami-shi, Niigata, Japan, 2011 (Ryo)	2 (7)	42 42	2 2	mature stage Sept 05	1	0.14 / 0.14 (0.14)	< 0.01/< 0.01 (< 0.01)	0.15/ 0.15 (0.15)	Koriyama JP2011C362B [Takashi, 2012a, report JP2011C362]
					3	0.12 / 0.12 (0.12)	< 0.01/< 0.01 (< 0.01)	0.13/ 0.13 (0.13)	
					7	0.09 / 0.09 (0.09)	< 0.01/< 0.01 (< 0.01)	0.10/ 0.10 (0.10)	
idem	2 (7)	41 41	3 3	early grain enlarement stage Aug 22	14	0.12 / 0.12 (0.12)	< 0.01/< 0.01 (< 0.01)	0.13/ 0.13 (0.13)	idem
					21	0.07 / 0.07 (0.07)	< 0.01/< 0.01 (< 0.01)	0.08/ 0.08 (0.08)	
82-9 Tanaka, Kuroishi-shi, Aomori, Japan, 2012 (Aomori Toyomaru)	2 (7)	50 50	3 3	shell enlargement stage Aug 29	1	0.14 / 0.13 (0.14)	< 0.01/< 0.01 (< 0.01)	0.15/ 0.14 (0.15)	Aomori JP2012C103A Yoshiyuki, 2013d, report JP2012C103]
					3	0.12 / 0.12 (0.12)	< 0.01/< 0.01 (< 0.01)	0.13/ 0.13 (0.13)	
					7	0.06 / 0.06 (0.06)	< 0.01/< 0.01 (< 0.01)	0.07/ 0.07	
idem	2 (7)	50 50	3 3	shell enlargement stage Aug 15	14	0.03 / 0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/ 0.04 (0.04)	idem
					21	0.02 / 0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/ 0.03 (0.03)	

Pulses

Soya bean, dry

A 50 g/L soluble concentrate of cyclaniliprole (4.5%) was applied on soya bean in field residue trials in Japan (six decline trials) [Takashi N, 2012b, report JP2011C361 and Yoshiyuki T, 2013e, report JP2012C102]. With an interval of 6–8 days two foliar spray applications of 38–49 g ai/ha were applied at a volume of 1500–1900 L/ha with a backpack sprayer. Trials were performed in the 2011 and 2012 growing seasons; sampling days ranged from 1–21 days. The samples were air dried in a greenhouse for 5–8 days, followed by threshing and subsequent sorting by wind in a winnowing basket. The results are summarised in Table 95.

Table 95 Supervised field trials on soya bean (dried) treated with two pre-harvest foliar spray formulations with cyclaniliprole (50SL) by back pack spraying

SOYA BEAN, DRIED Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
82-9 Tanaka, Kuroishi-shi, Aomori, Japan, 2011 (Oshuzu)	2 (7)	38 38	3 3	grain filling stage Oct 03	3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	Aomori JP2011C361A [Takashi, 2012b, report JP2011C361]
idem	2 (7)	38 38	3 3	grain filling stage Sept 29	7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	38 38	3 3	gain filling stage Sept 22	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	38 38	3 3	shell enlargement Sept 15	21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
Kuroishi, Shimanto-cho, Takaokagun Kochi, Japan, 2011 (Fukuyutaka)	2 (8)	45 45	3 3	mature stage Nov 12	3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	Kochi JP2011C361B [Takashi, 2012b, report JP2011C361]
	2 (7)	45 45	3 3	mature stage Nov 08	7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
	2 (7)	45 45	3 3	mature stage Nov 01	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	45 45	3 3	mature stage Oct 25	21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
Tsubone, Murakami-shi, Niigata, Japan, 2012 (Enrei)	2 (6)	46 46	3 3	time of harvesting Oct 24	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	NiigataJP2012C102A [Yoshiyuki, 2013d, report JP2012C102]
idem	2 (6)	46 46	3 3	proper time of harvesting Oct 22	3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (6)	46 46	3 3	immediately before harvesting Oct 19	7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (8)	46 46	3 3	middle leaf falling stage Oct 13	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
idem	2 (7)	46 46	3 3	leaf/middle yellowing stage of pods Oct 05	21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
Ogawa-machi, Hakusan-shi, Ishikawa,	2 (7)	48 48	3 3	mature stage Oct 08	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	Ishikawa JP2012C102B [Yoshiyuki, 2013d, report JP2012C102]

SOYA BEAN, DRIED Location, Country; year; (variety)	No. (int)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Japan, 2012 (Enrei)									
idem	2 (7)	48 48	3 3	leaf falling stage Oct 06	3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	48 48	3 3	leaf falling stage Oct 02	7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	48 48	3 3	grain enlargement stage Sept 25	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
idem	2 (7)	48 48	3 3	grain enlargement stage Sept 18	21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
2894-1, Naruto, Sanmu-shi, Chiba, Japan, 2012 (Sachiyutaka)	2 (7)	49 49	3 3	time of harvesting Oct 29	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	Chiba JP2012C102C [Yoshiyuki, 2013d, report JP2012C102]
idem	2 (7)	49 49	3 3	time of harvesting Oct 27	3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (8)	49 49	3 3	growth stage Oct 24	7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	49 49	3 3	growth stage Oct 16	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
idem	2 (7)	49 49	3 3	growth stage Oct 09	21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
Awano-cho, Ise-shi, Mie Prefecture, Japan, 2012 (Fukuyutaka)	2 (7)	40 40	3 3	mature stage Nov 14	1	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	Mie JP2012C102D [Yoshiyuki, 2013d, report JP2012C102]
idem	2 (7)	40 40	3 3	mature stage Nove 12	3	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	40 40	3 3	mature stage Nov 08	7	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (7)	40 40	3 3	Leaf falling stage Nov 01	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
idem	2 (6)	40 40	3 3	Leaf falling stage Oct 25	21	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	

Tree nuts

A 50 g/L soluble concentrate of cyclaniliprole (IKI-3106 50 SL aka IBE 4064) was applied on tree nuts in field residue trials in USA (five almond and five pecan) [Wiedmann & McDonald, 2014d, report IB-2012-JLW-019-01-01]. Three spray applications of 99–105 g ai/ha with an interval of 13–14-days were applied at in a volume of 1020–1700 L/ha. Trials were performed in the 2012 growing season and include one decline trial. Samples of hulls and nutmeat weighed 1.0–1.2 kg, except one duplicate sample of 0.7 kg in trial IB-2012-JLW-019-08 on day 25 and day 29, and were handpicked at mature stage from the ground around 4–10 trees. The results are summarised in Table 96.

Table 96 Supervised field trials on tree nuts treated with a formulation with cyclaniliprole (50SL) using a pre-harvest airblast sprayer

TREE NUTS (nut meat) Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Orland, CA, USA, 2012 (Non-Pareil) Almond	3 (14)	100 100 100	7 7 7	July 26	30	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-01 [Wiedmann &McDonald2014d]
Chico, CA, USA, 2012 (Non-Pareil) Almond	3 (14)	100 100 99	7 7 7	July 24	30	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-02 [Wiedmann &McDonald2014d]
Madera, CA, USA, 2012 (Non-Pareil) Almond	3 (13-15)	101 99 101	10 10 10	July 24	30	0.011/ 0.018 (0.015)	< 0.01/< 0.01 (< 0.01)	0.022/ 0.030 (0.026)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-03 [Wiedmann &McDonald2014d]
Strathmore, CA, USA, 2012 (Fritz) Almond	3 (14-15	99 100 100	6 6 7	Oct 05	31	0.015/ < 0.01 (0.013)	< 0.01/< 0.01 (< 0.01)	0.026/ < 0.01 (0.024)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-04 [Wiedmann &McDonald2014d]
Terra Bella, CA, USA, 2012 (Monterey) Almond	3 (14-15)	100 100 101	6 6 6	Oct 05	20	< 0.01/ 0.012 (0.011)	< 0.01/< 0.01 (< 0.01)	< 0.01/ 0.023 (0.022)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-05 [Wiedmann &McDonald2014d]
					25	0.015/ 0.010 (0.013)	< 0.01/< 0.01 (< 0.01)	0.026/ 0.021 (0.024)	
					31	0.013/ 0.013 (0.013)	< 0.01/< 0.01 (< 0.01)	0.024/ 0.024 (0.024)	
					39	< 0.01/ 0.011 (0.011)	< 0.01/< 0.01 (< 0.01)	< 0.01/0.022 (0.022)	
Anton, TX, USA, 2012 (Western	2 (13)	103 104	7 7	Oct 31	14	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-06 [Wiedmann

TREE NUTS (nut meat) Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Schley) Pecan									&McDonald2014d]
Pearsall, TX, USA, 2012 (Cheyenne) Pecan	3 (13-14)	102 102 103	8 8 8	Sept 20	29	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-07 [Wiedmann &McDonald2014d]
Alexandria, LA, USA, 2012 (Creek) Pecan	3 (14)	105 102 103	9 9 11	Oct 11	20	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-08 [Wiedmann &McDonald2014d]
					25	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
					29	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	
					40	< 0.01/< 0.01 (< 0.01)	0.022/< 0.01 (0.016)	0.033/< 0.01 (0.027)	
Bailey, NC, USA, 2012 (Stuart) pecan	3 (12-14)	103 101 99	9 7 7	Oct 10	30	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-09 [Wiedmann &McDonald2014d]
Girard, GA, USA, 2013 (Desirables) pecan	3 (14-15)	101 99 99	9 9 9	Sept 29	17	< 0.01/< 0.01 (< 0.01)	< 0.01/0.012 (0.011)	< 0.01/0.023 (0.021)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-10 [Wiedmann &McDonald2014d]

Infusions

A 50 g/L soluble concentrate of cyclaniliprole was applied on tea in field residue trials in Japan (6) [Koki M, 2012, report JP2012C133 and Yoshiyuki T, 2013f, report JP2012C101]]. One foliar spray application of 171–199 g ai/ha was applied at a volume of L/ha (302–350 L/10 are) with a knap sack sprayer. Trials were performed in the 2011 and 2012 growing seasons and include two decline trials; sampling days ranged from 3–21 days. Sample sizes ranged from 225–420 gram. The soil type in all trials was loam. The results are summarised in Table 97.

Table 97 Supervised field trials on tea treated with one pre-harvest foliar spray formulation with cyclaniliprole (50SL)

TEA Location, Country; year; (variety)	g ai/ha	g ai/hL	GS & last treatment day	Commodity	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
Naruto, Sanmu-shi Chiba, Japan, 2011 (Yabukita)	199	5	3-4 leaf, July 10	Crude tea	3	8.41/8.35 (8.38)	2.13/2.08 (2.10)	10.7/10.6 (10.6)	JPPA-Chiba, JP201C133A [Koki M, 2012]
				Hot water infusion	3	1.67 / 1.61 (1.64)	0.12 / 0.11 (0.12)	1.80/1.72 (1.76)	idem
idem	idem	idem	3-4 leaf, July 6	Crude tea	7	3.14 / 3.11 (3.13)	0.55 / 0.54 (0.54)	3.73/3.68 (3.70)	JPPA-Chiba, JP201C133A [Koki M, 2012]
				Hot water infusion	7	0.67 / 0.65 (0.66)	0.04 / 0.03 (0.04)	0.71/0.68 (0.70)	idem
idem	idem	idem	1-2 leaf, June 29	Crude tea	14	0.36 / 0.35 (0.36)	0.12 / 0.12 (0.12)	0.49/0.48 (0.48)	JPPA-Chiba, JP201C133A [Koki M, 2012]
				Hot water infusion	14	0.06/0.06 (0.06)	< 0.01 / < 0.01 (< 0.01)	0.08/0.08 (0.08)	idem
idem	idem	idem	sprouting, June 22	Crude tea	21	< 0.01/< 0.01 (< 0.01)	< 0.01 / < 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JPPA-Chiba, JP201C133A [Koki M, 2012]
				Hot water infusion	21	< 0.01 / < 0.01 (< 0.01)	< 0.01 / < 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
Niyodogawa-cho Mori, Agawa-gum Kochi, Japan, 2011 (Yabukita)	191	5	4 leaf, July 4	Crude tea	3	4.88 / 4.78 (4.83)	0.09 / 0.09 (0.09)	4.98/4.88 (4.93)	JPPA-Kochi, JP201C133B [Koki M, 2012]
				Hot water infusion	3	0.61/0.60 (0.61)	< 0.01 / < 0.01 (< 0.01)	0.63/0.62 (0.63)	idem
idem	idem		3-4 leaf, June 30	Crude tea	7	3.18 / 3.03 (3.10)	0.11 / 0.11 (0.11)	3.30/3.15 (3.22)	JPPA-Kochi, JP201C133B [Koki M, 2012]
				Hot water infusion	7	0.29 / 0.25 (0.27)	< 0.01 / < 0.01 (< 0.01)	0.31/0.27 (0.29)	idem
idem	idem	idem	2-3 leaf, June 23	Crude tea	14	0.46 / 0.45 (0.46)	0.31 / 0.30 (0.30)	0.79/0.77 (0.78)	JPPA-Kochi, JP201C133B [Koki M, 2012]
				Hot water infusion	14	0.05 / 0.05 (0.05)	< 0.01 / < 0.01 (< 0.01)	0.07/0.07 (0.07)	idem
Idem	idem	idem	1-2 leaf, June 15	Crude tea	21	< 0.01 / < 0.01 (< 0.01)	< 0.01 / < 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	JPPA-Kochi, JP201C133B [Koki M, 2012]
				Hot water infusion	21	< 0.01 / < 0.01 (< 0.01)	< 0.01 / < 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	idem
Kamiyaganuki, Iiruma-shi, Saitama, Japan, 2012	185	5	3-leaf, second grade tea leave, July 14	Crude tea	3	13.0 / 12.9 (13.0)	1.31 / 1.30 (1.31)	14.4/14.3 (14.3)	Saitama-Tea JP2012C101A [Yoshiyuki, 2013f]

TEA Location, Country; year; (variety)	g ai/ha	g ai/hL	GS & last treatment day	Commodity	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
						Parent	NK-1375	Tot.	
(Sayama-midori)									
				Hot water infusion		1.83 / 1.70 (1.77)	0.05 / 0.05 (0.05)	1.88/1.75 (1.82)	
Naruto, Sanmu-shi Chiba, Japan, 2012 (Yabukita)	174	5	3-4 leaf, opening, July 13	Crude tea	3	6.84 / 6.66 (6.75)	0.68 / 0.66 (0.67)	7.56/7.36 (7.46)	JPPA-Chiba JP2012C101B [Yoshiyuki, 2013f]
				Hot water infusion		1.27 / 1.22 (1.24)	0.03 / 0.03 (0.03)	1.30/1.25 (1.28)	idem
Niyodogawa-cho Mori, Agawa-gum Kochi, 2012 (Yakubita)	189	5	4-leaf stage, May 7	Crude tea	3	28.3 / 27.8 (28.0)	1.46 / 1.36 (1.41)	29.9/29.2 (29.6)	JPPA-Kochi JP2012C101C [Yoshiyuki, 2013f]
				Hot water infusion		2.72 / 2.67 (2.70)	0.04 / 0.04 (0.04)	2.76/2.71 (2.74)	idem
Nagasato, Chiran-cho, Minamikyusyu-shi Kagoshima, 2012 (Yamato-midori)	171	5	4-5 leaf stage, May 7	Crude tea	3	16.5 / 16.2 (16.4)	0.62 / 0.62 (0.62)	17.2/16.9 (17.0)	Kagoshima-Tea JP2012C101D [Yoshiyuki, 2013f]
				Hot water infusion		2.46 / 2.34 (2.40)	0.03 / 0.03 (0.03)	2.49/2.37 (2.43)	idem

Miscellaneous fodder and forage

Almond hulls

For description of the trials, see section on Tree nuts.

Table 98 Supervised field trials in almond (hulls) treated with a formulation with cyclaniliprole (50SL) using a pre-harvest airblast sprayer

ALMOND HULLS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
Orland, CA, USA, 2012 (Non-Pareil) almond hulls	3 (14,14)	100 100 100	7 7 7	July 26	clay loam	30	1.7 / 1.2 (1.5)	0.49 / 0.30 (0.39)	2.2/ 1.5 (1.9)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-01 [Wiedmann &McDonald2014d]
almond hulls, dry weight							2.1 / 1.4 (1.8)	0.60 / 0.36 (0.479)	2.8/ 1.8 (2.3)	idem

ALMOND HULLS Location, Country; year; (variety)	No, (interval)	g ai/ha	g ai/hL	GS & last treatment day	soil type	DALT (days)	Residues, mg/kg			Report; Trial no [ref]
							Parent	NK-1375	Tot.	
Chico, CA, USA, 2012 (Non-Pareil) almond hulls	3 (14,14)	100 100 99	7 7 7	July 24	clay	30	2.3 / 2.1 (2.2)	0.58 / 0.50 (0.54)	2.9/ 2.6 (2.8)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-02 [Wiedmann &McDonald2014d]
almond hulls, dry weight							2.6 / 2.4 (2.5)	0.67 / 0.57 (0.62)	3.3/ 3.0 (3.2)	
Madera, CA, USA, 2012 (Non-Pareil) almond hulls	3 (13,15)	101 99 101	10 10 10	July 24	loamy sand	30	2.7 / 2.9 (2.8)	0.69 / 0.71 (0.70)	3.4/ 3.6 (3.5)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-03 [Wiedmann &McDonald2014d]
almond hulls, dry weight							3.2 / 3.4 (3.3)	0.81 / 0.84 (0.83)	4.1 4.3 (4.2)	
Strathmore, CA, USA, 2012 (Fritz) almond hulls	3 (14,15	99 100 100	6 6 7	Oct 05	loam	31	1.5 / 1.6 (1.5)	0.21 / 0.26 (0.247)	1.7/ 1.9 (1.8)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-04 [Wiedmann &McDonald2014d]
almond hulls, dry weight							1.9/ 2.1 (2.0)	0.27 / 0.35 (0.31)	2.2/ 2.5 (2.3)	
Terra Bella, CA, USA, 2012 (Monterey) almond hulls	3 (14,15)	100 100 101	6 6 6	Oct 05	sandy loam	20	2.1 / 1.7 (1.9)	0.37 / 0.32 (0.34)	2.5/ 2.0 (2.2)	IB-2012-JLW-019-01-01 IB-2012-JLW-019-01-01-05 [Wiedmann &McDonald2014d]
						25	1.8 / 1.6 (1.7)	0.31 / 0.26 (0.29)	2.2/ 1.9 (2.0)	
						31	1.8 / 1.8 (1.8)	0.34 / 0.31 (0.32)	2.2/ 2.1 (2.2)	
						39	1.5 / 1.8 (1.6)	0.29 / 0.32 (0.31)	1.8/ 2.1 (2.0)	
almond hulls, dry weight						20	2.8 / 2.3 (2.5)	0.49 / 0.44 (0.47)	3.3/ 2.8 (3.0)	idem
						25	2.1 / 1.8 (2.0)	0.36 / 0.30 (0.33)	2.5/ 2.2 (2.3)	
						31	2.2 / 2.1 (2.1)	0.40 / 0.36 (0.38)	2.6/ 2.5 (2.5)	
						39	1.8 / 2.1 (2.0)	0.36 / 0.38 (0.37)	2.2/ 2.5 (2.3)	

FATE OF RESIDUES IN PROCESSING

In processing

The Meeting received information on the nature of residues under conditions simulating pasteurisation, baking/brewing/boiling and sterilisation. In addition, the Meeting received processing studies on apple, peach, plum, grape, tomato and tea. Processing studies included studies spiked samples and processing studies with incurred residues. The spiked processing studies serve to determine whether hydrolysis products, as found in the hydrolysis study, are formed in processed commodities. The processing studies, with incurred residues, serve for the derivation of processing factors.

Nature of residues under processing

The behaviour of cyclaniliprole was studied under conditions simulating pasteurisation, baking/brewing/boiling and sterilisation [Button, 2015, JSM0542]. Two radiolabelled forms of cyclaniliprole, ^{14}C-phenyl-labelled and ^{14}C-pyrazole-labelled, were used at an actual concentration of 0.097–0.106 mg/L. The aqueous buffer solutions (glacial acetic acid (pH 4.0 and pH5.0) or sodium dihydrogen orthophospohate (pH 6.0) in water adjusted to pH with sodium hydroxide) were incubated for 20 minutes at 90 °C (pH 4), 60 minutes at 100 °C (pH 5) or 20 minutes at 120 °C (pH 6). Incubation temperatures were within 5 °C of the target value. At the end of the incubation period, the solutions were analysed for total radioactivity content by liquid scintillation counting, and by reversed phase HPLC to identify and quantify cyclaniliprole and its radiolabelled degradation products. Identification was confirmed by normal phase TLC. The test solutions were analysed on the day of sampling.

The recovery of radioactivity from the buffer solutions was in the range 92.2–105.6% of applied radioactivity.

Degradation of cyclaniliprole increased with pH and temperature (see Table 99). Cyclaniliprole remained stable during pasteurisation (99% of applied radioactivity remaining). Cyclaniliprole degraded during baking/brewing/boiling (79–89% of applied radioactivity remaining) and sterilisation (53–65% of applied radioactivity remaining). The phenyl radiolabelled form degraded to BPQO and BCPBA, while the pyrazole radiolabelled form, degraded to YT-1327 and unknown component A (see Table 99).

Table 99 Quantification of radioactivity in buffer solutions following incubation

Component	pasteurisation; 20 min 90 C, pH 4 [% TAR]	baking/brewing/boiling 60 min 100 C, pH 5 [% TAR]	sterilisation 20 min 120 C, pH 6 [% TAR]
Phenyl-labelled cyclaniliprole			
Cyclaniliprole	98.6	87.0	64.7
Component A	0.9	1.7	-
BCPBA[a]	-	-	22.7
BPQO[b]	-	10.7	15.7
Others[c]	0.8	1.1	2.5
Total	100.3	100.5	105.6
Pyrazole-labelled cyclaniliprole			
Cyclaniliprole	99.8	79.2	52.7
YT-1327[d]	-	11.0	44.2
Component A	-	1.2	1.9
Others[c]	1.2	0.8	1.4
Total	101	92.2	100.2

[a] BCPBA: 3-bromo-5-chloro-2-((3-chloropyridin-2-yl)amino) benzoic acid

[b] BPQO: 4-bromo-2,6-dichloro-11H-pyrido[2,1-b]quinazolin-11-one

[c] Radioactivity not associated with specific components

[d] YT-1327: 3-bromo-N-(1-cyclopropylethyl)-1H-pyrazole-5-carboxamide

Processing studies with spiked samples

Apple, study 1

The purpose of this study was to quantify the magnitude of residues of cyclaniliprole and its metabolites NK-1375 and degradation products BPQO, BCPBA and YT-1327 in apple processed commodities as a result of spiking apple fruits (variety Braeburn) before the processing procedure [Schäufele, 2016a, SQ74KP].

Commercially available apple fruits were spiked with cyclaniliprole, equivalent to an application rate of 0.417 kg ai/ha. The fruits for spiking were placed on clean and flat ground, tightly packed, but only 1 fruit layer thick. The fruits were then sprayed with a calibrated boom sprayer which fully covered the fruit area and the application was started and ended at least 1 meter before and after the fruit area. After the spray has dried in a heated greenhouse for 1.5 hours, apples were processed to canned fruits, apple sauce, juice and jelly according to common industrial practices at reduced scale. Specimens were then stored frozen at the test sites prior to shipment to the analytical laboratory. All specimens were maintained frozen (≤-18 °C) from the date of sampling until the date of extraction for a maximum storage duration of 59 days.

Canned fruits (the weights in brackets are for the treated fruits)

Unwashed apples (10.21 kg) were peeled and peeled fruits (8.67 kg; peel, 1.36 kg) were cut into smaller pieces (sample of 5.92 kg), with core and stalks removed (peeled fruit without cores and stalks, 4.76 kg; core and stalks, 1.05 kg). Water (2.09 kg), ascorbic acid (4.7 g) and glucose syrup (0.84 kg) was added to the apple pieces (sample of 3.72 kg) and heated until boiling (pH of intermediate was 3.21 and 3.22, regulation with citric acid not necessary). The boiled intermediate was filled in jars, the jars were filled up with water (2.27 kg), closed and pasteurised 1–15 minutes at 95–98 °C. After cooling, canned fruits (3.32 kg; 6.22 kg corrected for sub fractionation) and syrup (2.04 kg) were sampled. The yield for canned fruit was 65%.

Apple sauce (the weights in brackets are for the treated fruits)

Unwashed apples were cut into halves, stalks removed, and apples (3.81 kg) were boiled (5–8 minutes; water was added until the apple pieces were covered, 4.63 kg). After boiling (3.41 kg apples), apples were sieved (2.92 kg; without cooking water, 4.70 kg) and sugar (0.12 kg) was added until a dry substance of 16.5% was reached. Ascorbic acid was added (0.002 kg, pH of intermediate sample was 3.41 and 3.40, no adjustment with citric acid needed) and the intermediate (sample of 2.02 kg; 1.26 kg sampled) was filled into jars. Jars were closed and pasteurised 1–14 minutes at 95–98 °C (1.26 kg apple sauce; 2.92 kg corrected for sub-fractionation). The yield for apple sauce was 77%.

Apple juice (the weights in brackets are for the treated fruits)

Unwashed apples (22.11 kg) were mashed in a masher and pressed to extract the raw juice. The intermediates were raw juice (14.76 kg) and wet pomace (5.62 kg). Raw juice (sample of 11.66 kg) was pasteurised at 83-88 °C for 1-2 minutes to obtain juice (11.19 kg; 14.17 kg corrected for sub-fractionation). Apple wet pomace (4.52 kg) was dried in an oven at 65 °C until a moisture content of 10% was achieved. Dried pomace (0.96 kg) was sampled. The yield for apple juice was 64%.

Apple jelly (the weights in brackets are for the treated fruits).

Raw apple juice (0.75 kg) was mixed with gelling sugar (1 kg), heated up until boiling and cooked for 4 minutes. Lemon juice (0.03 kg) was added and well mixed with the cooked product. Apple juice contributed 37% to the mixture (0.75/2.05 kg). Jelly (1.67 kg) was sampled after cooling down (1.67/2.05 kg = 81%).

Samples were analysed for cyclaniliprole, NK-1375, using LC-MS/MS method JSM0269 and degradation products BPQO, BCPBA and YT-1327 using LC-MS/MS method XR44SB. The procedural recoveries were within the acceptable range of 70–120% for each analyte, except for

BPQO in apple fruit syrup (128%) and in apple sauce, before pasteurization (123%). The results are summarised in the section on analytical methods. No cyclaniliprole or NK-1375, BPQO, BCPBA and YT-1327 were found in any of the untreated control specimens (each < 0.01 mg/kg).

Cyclaniliprole and NK-1375 levels can be observed in Table 100. Residues of NK-1375, BPQO, BCPBA and YT-1327 were below the LOQ of 0.01 mg/kg in all samples. Therefore, the results for BPQO, BCPBA and YT-1327 are not shown in a table.

Since the apples were spiked, the processing factors derived from this study cannot be taken into account for derivation of overall processing factors. The study confirms that the hydrolysis products identified in the nature of residue study are not formed during apple processing involving pasteurisation and 4 min cooking periods. Whether the hydrolysis products are actually formed during sterilisation was not investigated in this study.

Table 100 Residues of cyclaniliprole and NK-1375 after processing of apple

Processed commodity	Cyclaniliprole [mg/kg]	NK-1375 [mg/kg]	Tot.	PF[a] parent	PF[b] Tot.
Fruit without stems	0.58/0.48 (0.53)	< 0.01/< 0.01 (< 0.01)	0.54		
Peeled fruits	0.12	< 0.01	0.13	0.23	0.24
Peel	2.9	< 0.01	2.9	5.5	5.4
Core and stalks	0.20	< 0.01	0.21	0.38	0.39
Peeled fruit without core and stalk	0.085	< 0.01	0.096	0.16	0.18
Canned fruits	0.067	< 0.01	0.078	0.13	0.14
Fruit syrup	0.033	< 0.01	0.044	0.06	0.08
Stalks	3.2	< 0.01	3.2	6.1	6.0
Solids after sieving	2.5	< 0.01	2.5	4.7	4.6
Apple sauce, non-pasteurised	0.24	< 0.01	0.25	0.46	0.47
Apple sauce, pasteurised	0.12	< 0.01	0.13	0.24	0.25
Raw juice	0.073	< 0.01	0.084	0.14	0.15
Wet pomace	2.1	< 0.01	2.1	3.9	3.9
Dry pomace	5.0	< 0.01	5.0	9.5	9.3
Juice	0.086	< 0.01	0.097	0.16	0.18
Jelly	< 0.01	< 0.01	< 0.02	< 0.02	< 0.02

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

Peach, study 1

A processing study with peaches was conducted in 2015 in Germany [Schäufele, M., 2016b, report QK27SS]. The purpose of this study was to quantify the magnitude of residues of cyclaniliprole and NK-1375, BPQO, BCPBA and YT-1327 in peach processed commodities as a result of spiking peach fruits before the processing procedure. Peaches were spiked once with an SL formulation of cyclaniliprole at application rate equivalent to 0.40 kg ai/ha. The fruits were placed on clean and flat ground, tightly packed, but only 1 fruit layer thick. The fruits were then sprayed with a calibrated boom sprayer which fully covered the fruit area. After spiking the fruits were carried over into a heated greenhouse for drying. The drying duration was 22 hours and 15 minutes. The spiked specimens were transported to the processing facility at ambient conditions and were stored at cooled conditions until start of processing. The processing of the treated fruits started two days after the spiking and one day after the fruits were completely dry. Untreated and treated fruits were processed into canned fruits (pasteurised), puree (pasteurised), juice (pasteurised) and jam following common industrial processes at reduced scale.

RAC peach samples

Prior to processing, RAC peach samples were taken from each batch (in duplicate from the treated batch). The stones were removed and separated from the flesh before freezing the specimens at the processing test sites. The weight ratio of the fruits without stones—whole fruits was 0.868.

Canned fruits

Peaches (10.01 kg) were blanched for 3–5 minutes in 78–80 °C hot water. Specimens of blanched fruits and blanching water were taken. A subsample of 6.57 kg of the blanched fruits was peeled. The peeled fruits weighed 5.93 kg. Samples of peeled fruits and peels were taken. A subsample of 3.68 kg of peeled fruits were cut in halves or quarters. The stones were removed. Fruits without stones weighed 3.03 kg. Water, citric acid, ascorbic acid and glucose syrup were added to the peaches and heated until boiling (pH?). The boiled intermediate was filled in jars. These jars were filled up with the water used for cooking, closed and pasteurised 2–9 minutes at 95–98 °C. After cooling, canned fruits and syrup were sampled. Canned (and drained) fruits weighed 1.78 kg (9.87 kg corrected for sub-fractionation). The yield for canned fruit was 99%.

Purée

Peaches (10.05 kg) were cut into halves, stones removed and boiled in water (5–10 minutes). The fruits after boiling weighed 6.04 kg. After boiling, peaches were sieved. Solid parts after sieving were sampled. Sugar was added to the sieved peaches until a dry substance of 16.5–16.6% was reached. Ascorbic acid was added. Intermediate puree weighed 5.77 kg. Samples of intermediate puree before pasteurisation were taken. The rest was divided into two portions, one for the processing to puree (1.78 kg) and one for the processing to jam (2.00 kg). For the processing to puree, the intermediate puree was filled into jars. Jars were closed and pasteurised 2-9 minutes at 95–98 °C. The final puree weighed 1.77 kg (5.73 kg corrected for sub-fractionation). The pasteurised puree was sampled after cooling down. For the processing of jam the intermediate puree was boiled and a 5% pectin solution was added. The final jam weighed 2.11 kg (6.09 kg corrected for sub-fractionation). Samples of jam were taken. Yields for jam and puree were 61 and 57%, respectively.

Juice (nectar)

Peaches (19.75 kg) were cut into halves, stones removed and mashed in a masher. The mash was pressed to extract the raw juice. The intermediates were raw juice (6.19 kg) and wet pomace (8.60 kg) from which samples were taken. The remaining 4.14 kg of raw juice was pasteurised at 83–90 °C for 2 minutes. The pasteurised juice weighed 3.99 kg (5.97 kg corrected for sub-fractionation). Juice was sampled. The remaining 7.72 kg of wet pomace was dried in an oven at 60–65 °C until a moisture content of ≤ 10% was achieved. Dry pomace weighed 0.98 kg (1.12 kg corrected for sub-fractionation). Dry pomace was sampled. The yield for juice is 30%.

All specimens were frozen on the day of collection and maintained frozen (-18 °C) from the date of sampling until the date of extraction. The maximum storage duration for processed samples was 69 days.

The analytical method for detection of cyclaniliprole and NK-1375 was validated within study JSM0269 and for BPQO, BCPBA and YT-1327 in study XR44SB. The overall mean recovery for cyclaniliprole, NK-1375, BPQO, BCPBA and YT-1327 was 93, 97, 84, 90 and 96% respectively, showing the methodology to be working within its requirements on each occasion of sample analysis.

BPQO, BCPBA and YT-1327 were found to be below the limit of quantitation (LOQ = 0.01 mg/kg) in any of the treated specimens. Therefore, results on BPQO, BCPBA and YT-1327 are not reported in the Table 101.

Table 101 Residues of cyclaniliprole and NK-1375 after processing of peaches

Location, Country; year; (variety)	Treatment (g ai/ha)	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
			Parent	NK-1375	Tot.			
04668 Motter-witz Germany 2015 (Amber Crest)	402.1[d]	Fruit without stone	0.73/0.83 (0.78)	< 0.01/< 0.01 (< 0.01)	0.74/0.84 (0.79)			QK27SS QK27SS-01 [Schäufele, M., 2016b]
		Whole fruit (RAC)[c]	0.64/0.72 (0.68)	< 0.01/< 0.01 (< 0.01)	0.65/0.73 (0.69)			
		blanching water	0.24	< 0.01	0.25	0.35	0.36	
		blanched fruits	0.56	< 0.01	0.57	0.82	0.83	
		peeled fruits	0.038	< 0.01	0.05	0.06	0.07	
		peel	7.41	< 0.01	7.4	11	11	
		stones	0.05	< 0.01	0.061	0.07	0.09	
		canned fruit	0.03	< 0.01	0.041	0.04	0.06	
		fruit syrup	< 0.01	< 0.01	< 0.01	0.01	0.01	
		solid parts after sieving	3.5	< 0.01	3.5	5.1	5.0	
		puree, before pasteurisation	0.57	< 0.01	0.58	0.84	0.84	
		puree, after pasteurisation	0.54	< 0.01	0.55	0.79	0.80	
		jam	0.54	< 0.01	0.55	0.79	0.80	
		raw juice	0.53	< 0.01	0.54	0.78	0.78	
		wet pomace	1.4	< 0.01	1.4	2.0	2.0	
		dry pomace	5.1	< 0.01	5.1	7.5	7.4	
		pasteurised juice	0.52	< 0.01	0.53	0.76	0.77	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

[c] Calculated value, taking the weight ratio of flesh and whole fruits of 0.868 in consideration

[d] Calculated value

Tomato, study 1

A processing study with tomatoes was conducted in 2015 in Germany [Schäufele, M., 2016c HH97BD]. The purpose of this study was to quantify cyclaniliprole, its metabolite NK-1375, and its degradation products BPQO, BCPBA and YT-1327 in tomato processed commodities as a result of spiking tomato fruits before the processing procedure.

Tomatoes were treated with an SL formulation of cyclaniliprole once with a calculated application rate of 0.39 kg ai/ha. The spiking process was done using an exaggerate rate of cyclaniliprole. The fruits for spiking were placed on clean and flat ground, tightly packed, but only 1 fruit layer thick. The fruits were then sprayed with a calibrated boom sprayer which fully covered the fruit area and the application was started and ended at least 1 meter before and after the fruit area. Once the fruits were spiked and dried, the spiked specimens were transported to the processing facility at ambient conditions. One day after spiking, tomatoes were processed to canned fruits, ketchup, paste, juice and dried tomatoes. Prior to processing, RAC tomato samples were taken from each batch (in duplicate from the treated batch).

Canned tomatoes

Tomatoes (8.98 kg) were blanched for 1 minute in 82–85 °C hot water. Blanched tomatoes weighed 9.01 kg of which 6.52 kg was used for peeling. Peeled tomatoes and peels weighed 5.88 and 0.56 kg respectively. 2.52 kg of peeled tomatoes were put into jars and the jars filled up with tap water. Jars were closed and sterilised at 118–125 °C for 5–9 minutes in an autoclave. The final weight of the

canned tomatoes was 1.79 kg (5.77 kg corrected for sub-fractionation). The yield from RAC to canned tomatoes was 64%.

Juice

Tomatoes (18.22 kg) were crushed to tomato mash. The mash was heated to 80–87 °C for 30 minutes. The heated mash was pressed and sieved. The intermediate products were raw juice (14.23 kg) and wet pomace (2.73 kg).

A subsample of 2.51 kg of wet pomace was dried in an oven at 60–65 °C until a moisture content of ≤ 10% was achieved. Dry pomace weighed 0.33 kg (0.36 kg corrected for sub-fractionation).

Mashing, heating, pressing and sieving steps to obtain raw juice were common to tomato paste, puree, ketchup and juice processing. A subsample of 2.50 kg of raw juice was pasteurised at 92 °C for 5 minutes. The resulting pasteurised juice weighed 2.45 kg (13.95 kg corrected for sub-fractionation). The yield for tomato juice was 77%.

Tomato purée and ketchup

A subsample of 1.93 kg raw juice was used for tomato purée/ketchup processing. Raw tomato juice before pasteurization was concentrated by using a concentration plant (55 °C, vacuum) until a dry matter content of 10.6–11.6% was achieved. After the concentration, the purée (7–14% dry matter, weighing 0.87 kg) was sampled. The remaining 0.43 kg was used for seasoning. After seasoning with vinegar (max 0.4% relating to the dry matter content of the puree), sugar (min 42% relating to the dry matter content of the puree) and salt (max 15% relating to the dry matter content of the puree), the mixture was pasteurised at 92 °C for 5 minutes. The final ketchup weighed 0.45 kg (6.71 kg corrected for sub-fractionation). After cooling, ketchup was sampled. The yield from RAC to ketchup was 37%.

Paste

A subsample of 5.08 kg raw juice was used for tomato paste processing. Raw tomato juice before pasteurization was concentrated by using a concentration plant (53–55 °C, vacuum) until a dry matter content of 28.9–29.4% was achieved. The intermediate was pasteurised at 92 °C for 5 minutes. Tomato paste (25–35% dry matter) weighed 0.46 kg (1.29 kg corrected for sub-fractionation). After cooling down, tomato paste was sampled. The yield from RAC to tomato paste was 7%.

Dried tomatoes

Tomatoes (6.02 kg) were cut into slices. Slices were dried in an oven at 65 °C until a moisture content of ≤ 10% was achieved. Dried tomatoes weighed 0.40 kg. No sub-fractionation took place. The yield from RAC to dried tomatoes was 7%.

All processed commodities were stored frozen (target temperature of -18 °C) from the day of collection onwards for a maximum of 62 days.

Samples were analysed for parent cyclaniliprole, NK-1375, BPQO, BCPBA and YT-1327 using LC-MS/MS method JSM0269. Untreated control samples of RAC and processed commodities had residues below the LOQ of 0.01 mg/kg. The overall mean recovery for parent cyclaniliprole, NK-1375, BPQO, BCPBA and YT-1327 was 94, 91, 92, 96 and 92% respectively, showing the methodology to be working within its requirements on each occasion of sample analysis.

Parent cyclaniliprole NK-1375, BPQO, BCPBA or YT-1327 were found above the LOQ of 0.01 mg/kg in the untreated samples, with the exception of the dry pomace in which parent cyclaniliprole was detected at 0.012 mg/kg. The level of parent cyclaniliprole found in dry pomace from the untreated specimens was insignificant compared to the treated specimens.

The study shows that the degradation products BPQO, BCPBA and YT-1327 are not detected (< 0.01 mg/kg) in processed tomato products using spiked tomato samples treated with an exaggerated application rate except tomato paste (0.029, 0.021 and 0.022 mg/kg per degradation product,

respectively, compared to 1.74 mg/kg parent and < 0.01 mg/kg NK-1375, each degradation product accounting for < 2% of the total residue). The processing factors of 1.26 for tomato puree, 1.17 for ketchup, 3.28 for paste and 8.53 for dried tomatoes indicate that concentration of parent cyclaniliprole occurs in these commodities. The processing factor of < 0.02 for canned tomatoes and 0.70 for juice indicates that no concentration of parent cyclaniliprole occurs in those commodities.

Table 102 Residues of cyclaniliprole and NK-1375 in tomato after processing

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg [a]			PF[b] parent	PF[c] Tot.	Report; Trial no [ref]
				Parent [a]	NK-1375 [a]	Tot.[a]			
04668 Motterwitz Germany 2015 Tomatoes (variety not available)	0.3939 (n.a.)	1	Fruits (RAC)	0.59/0.46 (0.53)	< 0.01/< 0.01 (< 0.01)	0.60/0.47 (0.54)			HH97BD HH97BD-1 [Schäufele, M., 2016c].
		1	Blanching water	0.21	< 0.01	0.22	0.40	0.41	
			Blanched tomatoes	0.35	< 0.01	0.36	0.66	0.67	
			Peel	3.6	< 0.01	3.6	6.8	6.7	
			Peeled fruits	< 0.01	< 0.01	< 0.012	< 0.02	< 0.02	
			Canned tomato	< 0.01	< 0.01	< 0.01	< 0.02	< 0.02	
			Vegetable stock	< 0.01	< 0.01	< 0.01	< 0.02	< 0.02	
			Raw juice	0.41	< 0.01	0.42	0.77	0.78	
			Wet pomace	2.8	< 0.01	2.8	5.2	5.2	
			Dry pomace	12	0.02	12	22	21	
			Puree	0.67	< 0.01	0.68	1.26	1.3	
			Ketchup	0.62	< 0.01	0.63	1.17	1.2	
			Paste	1.7	< 0.01	1.7	3.3	3.2	
			Juice	0.37	< 0.01	0.38	0.70	0.70	
			Dried tomatoes	4.5	< 0.01	4.5	8.5	8.4	

[a] Residues expressed in mg cyclaniliprole (IKI-3106)/kg and mg NK1375/kg, respectively and values are presented as single and (means) of field samples

[b] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[c] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

Table 103 Degradation products BPQO, BCPBA and YT-1327 in tomato after processing

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			Report; Trial no [ref]
				BPQO	BCPBA	YT-1327	
04668 Motterwitz Germany 2015 Tomatoes (variety not available)	0.3939 (n.a.)	1	Fruits (RAC)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	HH97BD HH97BD-1 [Schäufele, M., 2016c].
		1	Blanching water	< 0.01	< 0.01	< 0.01	
			Blanched tomatoes	< 0.01	< 0.01	< 0.01	
			Peel	< 0.01	< 0.01	< 0.01	
			Peeled fruits	< 0.01	< 0.01	< 0.01	
			Canned tomato	< 0.01	< 0.01	< 0.01	
			Vegetable stock	< 0.01	< 0.01	< 0.01	
			Raw juice	< 0.01	< 0.01	< 0.01	
			Wet pomace	< 0.01	< 0.01	< 0.01	
			Dry pomace	< 0.01	< 0.01	< 0.01	
			Puree	< 0.01	< 0.01	< 0.01	

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			Report; Trial no [ref]
				BPQO	BCPBA	YT-1327	
			Ketchup	< 0.01	< 0.01	< 0.01	
			Paste	0.029	0.021	0.022	
			Juice	< 0.01	< 0.01	< 0.01	
			Dried tomatoes	< 0.01	< 0.01	< 0.01	

Processing studies with incurred residues

Apple, study 2

The purpose of this study was to quantify the magnitude of cyclaniliprole and its metabolite NK-1375 at harvest in apples raw agricultural commodities (RAC) and processed commodities as a result of two actual applications at 0.038-0.043 kg ai/ha (plot 2), or at 0.12–0.13 kg ai/ha (plot 3; only one trial) applied on a 13–14-day interval with a pre-harvest interval (PHI) of 13–14-days [Schäufele, 2013b report JSM0348]. Four residue trials in Germany, France, and Italy were conducted in 2012. Apples were processed to canned fruits (according to ANADIAG SOP PO 0356), puree (according to ANADIAG SOP PO 0369), and apple juice (according to ANADIAG SOP PO 0268), mimicking common industrial processes. The processing of apples started 3 days after being collected in the field the latest. The specimens for processing purposes were delivered at ambient conditions at the processing facility. The specimens for processing purposes were stored at ambient conditions for a maximum of 2 days until processing. Processed commodity specimens were stored frozen (-18 °C) for a maximum of 45 days until analysis.

Samples were analysed for parent cyclaniliprole and metabolite NK-1375 using LC-MS/MS analytical method JSM0269. The average procedural recovery was within the acceptable range of 70–120% for each analyte. Procedural recoveries were only conducted with the RAC. No cyclaniliprole or NK-1375 was found in untreated control samples (< 0.01 mg/kg).

Canned fruits

Unwashed RAC fruits (approximately 3 kg; 5 kg for the balance study) were peeled and submerged in boiling water for approximately 30–60 seconds to avoid enzymatic browning (blanching). The peeled and blanched fruits were cut in quarters, the cores were removed. The quarters without cores (sample of 500 g) were placed into clean glass jars with syrup (500 g; water + sugar). To prevent browning and to correct the pH (value not reported), ascorbic acid was added. The jars were closed and sterilised at 120 °C for 20 minutes. By omission of the protocol requirements, the pH values of the final process products were not recorded, except for apple juice obtained from trial JSM0348-01. At the end of the process, the weight of the fruits after draining was 331–485 g (2029–2968 g; 3582–3807 g, corrected for sub-fractionation). The yield range was 66–97% and 71–76% in the balance study.

Apple puree

Unwashed RAC fruits (approximately 3 kg; 5 kg for the balance study) were cut into small pieces and cooked with 10% sugar (approximately 300 g) for 15 minutes at 85 °C. The mixture obtained was crushed and refined by passing through a fine sieve to separate peel and core residue. The puree (2360–2771 g; 4358–4416 g) was pasteurised at 90 °C for 2–3 minutes, pH value after processing not reported. No sub-fractionation took place. The yield range was 74–92% and 87% (balance study), respectively.

Apple juice

RAC samples (approximately 5 kg; 8 kg for the balance study) were crushed without washing or peeling. Fruits were introduced into a vertical press to separate the juice from pulp and peel (pomace). Ascorbic acid (0.75–0.9 g) was added to the juice to avoid any oxidation (pH was not reported).

Pectinolytic enzymes were added to the raw juice for decantation. After 12-24 hours at approximately 4 °C the juice (2032–2769 g) was racked and pasteurised for 2–5 minutes (instead of the 1 minute according to protocol) at 95 °C. No sub-fractionation took place. The yield range of clear juice versus RAC was 40–54% and 30–32% in the balance study. The pH after pasteurisation process was 3.8.

Apple pomace (only in the balance study)

After the pressing (see procedure for apple juice, balance study), the wet pomace cake (3851–4154 g, no sub-fractionation) was broken up to obtain the wet pomace sample (2800–3050 g). The yield ranges from RAC (8011–8045 g) to wet pomace cake (3851/8045 and 4154/8011 g) was 48–52%. Wet pomace is dried at 60 °C for 48 hours to obtain dry pomace (490 g). The yield range from wet pomace to dry pomace was 16–18%.

No parent cyclaniliprole or its metabolite NK-1375 were detected in any of the untreated apple specimens (each < 0.01 mg/kg). Cyclaniliprole in canned apples, apple puree and apple juice was below the limit of quantification (< 0.01 mg/kg), while in peels and cores, wet pomace and dry pomace quantifiable levels of cyclaniliprole were found (see Table 104). Residues of NK-1375 were only found at quantifiable levels in dry pomace.

Table 104 Residues of cyclaniliprole and NK-1375 after processing of apple

Location, Country; year; (variety)	Treatment (actual application rate in kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg [a]			PF[b] parent	PF[c] Tot.	Report; Trial no [ref]
				Parent [a]	NK-1375 [a]	Total [a]			
Impflingen, Germany, 2012 (Gala)	0.039 + 0.038 (13)	14	fruit	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01			JSM0348; JSM0348-01; [Schäufele M, 2013b] see section residue trials
			canned fruit	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			puree	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			juice	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
Brumath, France (N), 2012 (Jonagored)	0.042 + 0.043 (14)	14	fruit	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.031			JSM0348; JSM0348-02; [Schäufele M, 2013b] see section residue trials
			canned fruit	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			puree	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			juice	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
Grenade sur Garonne, France (S), 2012 (Golden)	0.040 + 0.040 (14)	13	fruit	0.01/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.02/0.03 (0.03)			JSM0348; JSM0348-03; [Schäufele M, 2013b] see section residue trials
			canned fruit	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			puree	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			juice	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	

Location, Country; year; (variety)	Treatment (actual application rate in kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg [a]			PF[b] parent	PF[c] Tot.	Report; Trial no [ref]
				Parent [a]	NK-1375 [a]	Total [a]			
San Sebastiano Curone, Italy, 2012 (Galaxy)	0.042 + 0.042 (14)	13	fruit	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01			JSM0348; JSM0348-04; [Schäufele M, 2013b] see section residue trials
			canned fruit	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			puree	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			juice	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
	0.128 + 0.125 (14)	14	fruit	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01	- [d]	- [d]	
			peeled and cored fruits	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			peels and cores	0.03	< 0.01	0.04	- [d]	- [d]	
			canned fruit	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			puree, before pasteurisation	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			puree, after pasteurisation	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			wet pomace	0.02	< 0.01	0.03	- [d]	- [d]	
			dry pomace	0.06	0.02	0.08	- [d]	- [d]	
			raw juice	< 0.01	< 0.01	< 0.01	- [d]	- [d]	
			juice	< 0.01	< 0.01	< 0.01	- [d]	- [d]	

[a] Residues expressed in mg cyclaniliprole (IKI-3106)/kg and mg NK1375/kg, respectively and values are presented as single and (means) of field samples

[b] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[c] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

[d] Since the RAC contained residues <LOQ, no reliable PF can be calculated.

Apple, study 3

A processing study with apples (variety Romes) was conducted in 2012 in the USA [Wiedmann & McDonald, 2013b, IB-2012-JLW-020-01-01]. The results for the part of the study in which residues of cyclaniliprole and NK-1375 in RAC have been investigated, have been evaluated in the section with supervised residue trials (additional plot in North Rose, New York, USA).

Cyclaniliprole was applied 3 times at an exaggerated application rate of 0.995–1.008 kg ai/ha and a 14-day interval to apples (first application at 24 Aug 2012, 0.995 kg ai/ha; second application at 7 Sept 2012, 0.1008 kg ai/ha; third application at 21 Sept 2012, 0.999 kg ai/ha). Apples were harvested 7 days after the last application and were processed to wet pomace and apple juice to simulate commercial practice as closely as possible, though processed by batch instead of continuously.

Apple juice (the weights in brackets are for the treated fruits)

Apples (47.6 kg) were mashed with a hammer mill. The mash (46.5 kg) was layered into cloth stacks on a hydraulic press and pressed to separate juice from wet pomace. While the mash was pressed, juice was collected (30.8 kg). No sub-fractionation took place, resulting in a yield of 65% from RAC to juice.

Apple pomace

After pressing, the wet pomace stacks were broken up, combined, mixed and wet pomace collected (14.5 kg), resulting in a yield of 30% from RAC to wet pomace.

Samples were maintained under frozen conditions (≤-11 °C). The storage intervals for the samples ranged from 127–179 days (processing time to time of extraction).

Samples were analysed for parent cyclaniliprole and metabolite NK-1375 using LC-MS/MS analytical method JSM0269. The average procedural recovery was within the acceptable range of 70-120% for each analyte, except for one recovery in apple juice (121%). No cyclaniliprole or NK-1375 was found in untreated control samples (each < 0.01 mg/kg?). Cyclaniliprole and NK-1375 levels can be observed in Table 105.

Table 105 Residues of cyclaniliprole and NK-1375 after processing of apple

Location, Country; year; (variety)	Treatment (g ai/ha, (interval))	DALT	Processed commodity	Cyclaniliprole [mg/kg]	NK-1375 [mg/kg]	Tot.	PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
North Rose, NY, USA, 2012 (Rome)	995 1010 999 (14)	7	Apple	0.79 / 0.72 / 0.61 (0.71)	0.082/ 0.087 /0.065 (0.078)	0.88/ 0.81 / 0.68 (0.79)			IB-2012-JLW-020 IB-2012-JLW-020-01 [Wiedmann &McDonald, 2013b] see section residue trials
			Apple juice	0.090/ 0.097 (0.094)	< 0.01/< 0.01 (< 0.01)	0.10/0.11 (0.10)	0.13	0.13	
			Wet pomace	2.3 / 2.2 (2.2)	0.24 / 0.26 (0.25)	2.6 / 2.5 (2.5)	3.2	3.2	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

Plums, study 1

A processing study with plums was conducted in 2013 (Madera, CA, USA) [Wiedmann & McDonald, 2013c, report IB-2013-JLW-005-01-01]. An SL formulation of cyclaniliprole was applied 3 times at exaggerated application rates of 0.974–0.985 kg ai/ha and a 7-day interval to plums. Plums were harvested 7-days after the last application and were processed to dried prunes using common industrial practices. Plums were sampled in duplicate. Fresh samples (RAC) were stored in field coolers on blue ice and transported directly from the field to the lab the same day of sampling.

Dried prunes

Fresh plums (2.40 and 2.47 kg) were placed in a dehydrator oven for 5 days resulting in dried prunes ((1.09 and 1.06 kg, respectively). No sub-fractionation took place. The yield range was 43–45%. Dried prune samples were packed in blue ice for delivery to the lab the same day of sampling.

The maximum storage duration for samples was 27 days. Samples were analysed for cyclaniliprole and NK1375 using LC-MS/MS method JSM0269. The average procedural recovery ranged from 86–102% at 0.01–1.0 mg/kg for plums and 74–108% at 0.01–4.7 mg/kg for prunes for both analytes, thus demonstrating the repeatability of the method on the days of analysis. Control samples of plums and prunes had no detectable parent cyclaniliprole or NK-1375 (each < 0.01 mg/kg). Parent cyclaniliprole and NK-1375 in treated samples of plums and dry prunes and processing factors for the dry prunes are summarised in Table 106.

The processing factor of 3.7 for prunes indicates that concentration of residues of cyclaniliprole occurs.

Table 106 Residues of cyclaniliprole and NK-1375 after processing of plums

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent [a]	NK-1375 [a]	Tot. [a]			
Madera, CA, USA, 2013 (French – prune type)	0.976 0.985 (7) 0.974 (7)	7	Plum	0.81/0.67 (0.74)	0.16/0.11 (0.13)	0.97/0.79 (0.88)			IB-2013-JLW-005 IB-2013-JLW-005-19 [Wiedmann &McDonald, 2013c] see section residue trials
			Dried prunes	2.4/3.1 (2.8)	0.41/0.55 (0.48)	2.9/3.7 (3.3)	3.7	3.7	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

Peach, study 2

A processing study with peaches was conducted in 2012 (Germany, the Netherlands, France (South) and Italy) [Schäufele, M., 2013i, report JSM0352]. An SL formulation of cyclaniliprole was applied 2 times at application rates of 0.0396–0.0429 kg ai/ha and in trial JSM0352-04 plot 3 at an exaggerated rate of 2 times 0.1220–0.1232 kg ai/ha and a 13–15-day interval to peaches.

Peaches for processing purposes were obtained from the untreated (plot 1) and the treated plots (plot 2 for "follow-up" and plot 3 (for "balance") at 13 DALA. Each sample consisted of whole peaches (flesh and stone) weighing more than 35 kg and were transported at ambient conditions. The stones were removed and separated from the flesh before freezing the specimens at the processing test sites. The weight ratio of the fruits without stones—whole fruits was determined and recorded (range 0.886 and 0.951, median 0.915). Processing started within 2 days after being collected.

Samples for "follow-up" processing (plot 2) were processed into canned fruits (sterilized), puree (pasteurized) and juice (pasteurized nectar) and for the balance study (plot 3) in addition various intermediate, side and end products were collected.

Canned fruits

Peaches (2.92–4.11 kg) were submerged in boiling water for approximately 30 to 60 seconds, and then placed immediately in cold water for 20 seconds to crack the peels. Fruits were cut in halves and the stones separated from the flesh (1.97–3.27 kg). Peels, flesh and peeling water were sampled. A subsample of 0.630–0.650 kg flesh halves were placed into clean glass jars with syrup (water + sugar; total weight of fruits +syrup: 1000g). The jars were closed and sterilised at 115 °C for 20 minutes in an autoclave. Sterilised canned peaches were sampled. Sterilised fruits after draining weighed 0.461–0.595 kg (1.52–2.95 kg corrected for sub-fractionation). The yield for canned fruits was 52–72%.

Purée

Peaches (3.06–4.06 kg) were submerged in boiling water for approximately 30 to 60 seconds, and then placed immediately in cold water for 20 seconds to crack the peels. Fruits were cut in halves and the stones separated from the flesh. Flesh halves were cut into small pieces. After preparation, fruits were crushed and cooked with 10% sugar for 5 minutes at 93–96 °C. The mixture obtained was passed through a vegetable mill and pasteurised at 95 °C for 5 minutes to get pasteurised puree (2.25–3.20 kg). No sub-fractionation took place. Puree was sampled before and after pasteurisation. The yield range for purée after cooking was 70–79% without correction for added sugar (ca. 10%).

Juice (nectar)

Peaches (3.50–5.40 kg) were submerged in boiling water for approximately 30 to 60 seconds, and then placed immediately in cold water for 20 seconds to crack the peels. Fruits were cut in halves and the stones separated from the flesh. Flesh halves were cut into small pieces, steamed at 85 °C for 5 minutes followed by crushing to obtain a fine purée. This purée was passed successively through two sieves to separate solid parts. The sieved purée was collected, (2.37–3.49 kg). Samples were taken of crushed fruits and solid parts. The yield range for purée in the juice process was 65–78% (no sugar added at this stage in this process).

A subsample of 1.83–2.61 kg of sieved puree was used for the next processing step. The purée's texture was adjusted by water addition (max. 40% of the final weight) and sugar addition (max. 20% of the final weight). The resulting nectar was stabilised by pH adjustment to 3.7–3.9 with citric acid. The nectar was canned, pasteurised at 95 °C for 5 minutes and cooled. The nectar weighed 3.02–4.31 kg (3.89–5.75 kg corrected for sub fractionation). Samples were taken of juice (nectar) before and after pasteurisation.

The yield range from RAC to juice was 106–129% (not corrected for added water (ca. 30% of total) and added sugar (ca. 9% of total).

All specimens were frozen on the day of collection and maintained frozen (-18 °C) from the date of sampling until the date of extraction. The maximum storage duration for processed samples was 170 days.

The analysis was conducted using LC-MS/MS method JSM0269. The overall mean concurrent recovery for cyclaniliprole was 90% and for NK-1375 it was 88%, showing the methodology to be working within its requirements on each occasion of sample analysis.

No parent cyclaniliprole and its metabolite NK-1375 were found in any of the untreated processed commodities from any of the trials (each < 0.01 mg/kg). In the puree specimen (untreated?) obtained from trial JSM0352-02 in the Netherlands parent cyclaniliprole residues of 0.09 mg/kg were detected in the first analysis, the re-analysis and the appropriate retain specimen showed no residues. Therefore, it must be concluded that the first analysis of the ship specimen was a mistake and there were indeed no residues within this puree specimen.

Parent cyclaniliprole and NK-1375 in treated samples of peach commodities are summarised in Table 107. NK-1375 was below the limit of quantification (LOQ = 0.01 mg/kg) in all samples except for peaches treated at the exaggerated rate with residues of 0.03 mg/kg NK-1375 in whole fruit? and 0.04 mg/kg NK-1375 in peach peel. The resulting lowest processing factors for parent cyclaniliprole were < 0.14 for canned peaches, purée and juice (nectar).

Table 107 Residues of cyclaniliprole and NK-1375 after processing (follow-up studies) of peaches

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
47918 Tonisvorst Germany 2012 (Revit)	0.0406 0.0428 (13)	13	Fruit without stone	0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.02 (0.03)			JSM0352 JSM0352-01 [Schäufele, M., 2013i] see section residue trials
			Whole fruit (RAC)[c]	0.02/0.01 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.02 (0.03)			
			canned fruit	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			Puree	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			nectar pasteurised	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
6086 Neer The Netherlands 2012 (Revita)	0.0412 0.0396 (15)	13	Fruit without Stone	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/0.02 (0.02)			JSM0352 JSM0352-02 [Schäufele, M., 2013i] see section
			Whole fruit (RAC)[c]	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.02/0.02 (0.02)			
			canned fruit	< 0.01	< 0.01	< 0.01	<1	< 0.5	
			Puree	< 0.01	< 0.01	< 0.01	<1	< 0.5	

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
			nectar pasteurised	< 0.01	< 0.01	< 0.01	<1	< 0.5	residue trials
31330 Grenade sur Garonne France (S) 2012 (Ronistar)	0.0429 0.0424 (14)	13	Fruit without Stone	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.03 (0.03)			JSM0352 JSM0352-03 [Schäufele, M., 2013i] see section residue trials
			Whole fruit (RAC)[c]	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.03 (0.03)			
			canned fruit	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			Puree	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			nectar pasteurised	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
15059 Volpedo Italy 2012 (Cresthaven)	0.0407 0.0399 (14)	13	Fruit without stone	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.03 (0.03)			JSM0352 JSM0352-04 [Schäufele, M., 2013i] see section residue trials
			Whole fruit (RAC)[c]	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.03 (0.03)			
			canned fruit	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			Puree	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
			nectar pasteurised	< 0.01	< 0.01	< 0.01	< 0.5	< 0.33	
	0.1220 0.1232 (14)	13	Fruit without stone	0.09/0.07 (0.08)	0.03/0.02 (0.03)	0.12/0.09 (0.11)			JSM0352 JSM0352-04 [Schäufele, M., 2013i] see section residue trials
			Whole fruit (RAC)[c]	0.08/0.06 (0.07)	0.03/0.02 (0.03)	0.11/0.08 (0.10)			
			Peel	0.09	0.04	0.13	1.3	1.3	
			peeling water	0.06	< 0.01	0.07	0.86	0.7	
			peeled fruits	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			canned fruit	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			puree, before pasteurisation	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			puree, after pasteurisation	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			crushed fruits	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			solid parts	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			nectar before pasteurisation	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	
			nectar after pasteurisation	< 0.01	< 0.01	< 0.01	< 0.14	< 0.1	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

[c] Calculated value, taking the weight ratio of flesh and whole fruits in consideration.

Grapes, study 1

A processing study with grapes was conducted in 2012 (Germany, France, Italy and Spain) [Schäufele, M., 2013n, JSM0350]. An SL formulation of cyclaniliprole was applied 2 times at application rates of 0.0322–0.0391 kg ai/ha and in two trials at exaggerated rates of 2 times 0.1005–0.1077 kg ai/ha and a 14–15 day interval to grapes.

RAC specimens were collected from the trials at 27–29 days after the last application (DALA), except in trial JSM0350-05. From this trial specimens were collected at 23 DALA due to the risk of rotten crop at 28±1 DALA. Specimens for processing purposes were transported to the processing laboratory at ambient conditions. Two specimens for processing purposes were collected from each plot. Both specimens consisted of wine grape bunches. One specimen weighed at least 75 kg and was processed into grape juice, young wine and stored wine. The other specimen weighed at least 8 kg and was processed into raisins. Immediately before the start of processing, raw agricultural

commodities were obtained from each specimen for processing purposes. From untreated plot 1 only 1 specimen was obtained, from each treated plot 2 and 3 two independently composite specimens of wine grape bunches were collected in a separate collection run. The processing of wine and juice started 2 days after being collected in the field the latest. The processing of raisins started 3 days after being collected in the field the latest.

Crushing and destemming

Grapes (72.44–78.45 kg) were crushed and de-stemmed by machine. Stemmed grapes weighed 68.66–74.86 kg of which subsamples were used for red juice and red wine processing.

Red juice processing

A subsample of the stemmed grapes (8.61–19.17 kg) was used for red juice processing. After addition of sulphites the must was heated at 70 °C during 20 minutes for colour extraction. The must was pressed to separate the raw juice from the pomace. After addition of pectolytic enzyme, the juice was stored at approximately 4 °C during 24h for clarification. Clear juice was separated from dregs. The clear juice weighed 5.62–13.17 kg (42.02–48.02 kg corrected for sub-fractionation). The clear juice was pasteurised at 80–85 °C during 10 minutes.

In the follow-up studies pasteurised juice was sampled. In the balance study stems after stemming, must after pressing/before heating, must after heating, wet pomace after pressing, raw juice after pressing, dregs after clarification, clear juice after clarification and juice after pasteurisation were sampled.

Red wine

A subsample of the stemmed grapes (50.37–60.60 kg) was used for red wine processing. Must was transferred in fermentation vat with addition of sulphites and yeasts. During the alcoholic fermentation (AF) density and temperature were monitored daily. A punching was also done daily. After the alcoholic fermentation, drop wine was removed and wet pomace was pressed. Drop wine and pressed wine together weighed 38.82–48.81 kg of which 37.79–48.81 kg were put together in a fermentation vat with addition of bacteria for malolactic fermentation (MLF). At the end of the malolactic fermentation, the wine was racked with addition of sulphites. Lees were removed. The MLF wine weighed 36.89–41.80 kg. 35.77–48.81 kg of wine was put in a vat for cold stabilisation. After cold stabilisation (min. 10 days at 4 °C), wine was clarified, sediments were separated, and wine was bottled with an addition of sulphites for stabilisation. The young wine obtained weighed 37.93–46.35 kg (47.60–57.26 kg corrected for sub-fractionation). The bottled wine was stored for 6 months (conditions not further specified). The yield range from RAC to red wine was 66–72%. In the balance study a portion of the wet pomace (1.20 kg) was dried at 60 °C (duration and pH not reported) to obtain dry pomace (0.471 kg). The yield range from wet pomace to dry pomace is 39%.

Juice (white)

Grapes (63.10–86.79 kg) were destemmed and pressed. The raw juice after pressing weighed 36.54–58.90 kg, of which 36.41–50.90 kg was used for clarification. Sulphites were added to raw juice. Pectolytic enzymes were added to the juice for clarification. The juice was stored at approx. 20 °C during 24h for clarification. Clear juice is separated from dregs (pH not reported). Cleared juice weighed 32.86–50.79 kg. The yield range from RAC to juice (white) was 52–62%.

In the balance study a portion of the wet pomace 1.58 kg was dried at 60 °C to obtain dry pomace. Weight of dry pomace after drying was not recorded.

Pressing and clarification steps were common to both wine and juice processing. Then one part of the cleared juice (3.0–4.6 kg) was pasteurised at 80–85 °C during 10 minutes to obtain pasteurised juice.

Wine (white)

The remaining part of the cleared juice (28.84–46.17 kg) was put into alcoholic fermentation vat for white wine processing. During the alcoholic fermentation density and temperature were monitored daily. Once the alcoholic fermentation was finished, the wine was racked with addition of sulphites. Lees were removed. After cold stabilisation (min. 10 days at 4 °C), wine was clarified, sediments were separated, and wine was bottled with an addition of sulphites for stabilisation. Young wine obtained weighed 24.33–41.22 kg (28.99–45.34 kg corrected for sub-fractionation). Bottled wine was stored for 6 months (conditions were not reported). The yield range from RAC to red wine was 48–55%.

Raisins

Grapes (5.00–5.99 kg) were de-stemmed manually. The berries weighed 4.66–5.64 kg of which 4.39–5.64 kg was soaked in an alkaline aqueous 2.5% potassium carbonate (K2CO3) and 2% ethyl oleate solution. After steeping, the berries were drained then dried in a drier at approximately 40 °C, in order to reach final water content between 13% and 23%. Raisins weighed 0.88–1.21 kg (1.07–1.21 kg corrected for sub-fractionation).

In the follow-up studies raisins were sampled. In the balance studies berries before dipping, dipping solution after dipping and raisins after drying were sampled. The yield range from RAC to raisins was 18–22%.

All specimens (RAC and processed commodity specimens) were frozen (-18 °C) on the day of collection and maintained frozen throughout storage and transport to the analytical laboratory.

The maximum storage duration for processed samples was 146 days for the “ship” specimens and 341 days for retain specimens of raw juice, clear juice, wet and dry pomace from trial JSM0350-4.

The analysis was conducted using analytical methods that had been previously validated [JSM0269]. The levels of recovery from fortified samples which were analysed along with the test samples ranged from 76 to 106% for recovery of cyclaniliprole and ranged from 75 to 108% for recovery of metabolite NK-1375, thus demonstrating the repeatability of the method on the days of analysis.

No cyclaniliprole and its metabolite NK-1375 were detected above the limit of detection in any of the RAC untreated wine grape bunch specimens from any of the trials.

Table 108 Residues of cyclaniliprole and NK-1375 after processing (balance study) of grapes

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
54200 Bruley France, 2012 Auxerrois (white variety)	0.1005 0.1066 (14)	29	Wine grape bunches prior to juice/wine processing (RAC)	0.17/0.11 (0.14)	0.02/0.01 (0.02)	0.19/0.12 (0.16)			JSM0350 JSM0350-2 [Schäufele, M., 2013n]
			Stems [j]	0.55	0.04	0.59	3.93	3.69	
			Must [j]	0.09	< 0.01	0.10	0.64	0.63	
			Wet pomace [j]	0.23	0.03	0.26	1.64	1.63	
			Dry pomace [j]	0.43	0.07	0.50	3.07	3.13	
			Dregs [j]	0.29	0.04	0.33	2.07	2.06	
			Clear juice [j]	0.06	< 0.01	0.07	0.43	0.44	
			Juice, after pasteurisation [j]	0.05	< 0.01	0.06	0.36	0.38	
			Stems [w]	0.62	0.04	0.66	4.43	4.13	
			Must [w]	0.09	< 0.01	0.10	0.64	0.63	
			Wet pomace [w]	0.24	0.04	0.28	1.71	1.75	

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
			Dry pomace [w]	0.43	0.07	0.50	3.07	3.13	
			Dregs [w]	0.27	0.04	0.31	1.93	1.94	
			AF wine [w]	0.05	< 0.01	0.06	0.36	0.38	
			Lees [w]	0.35	0.03	0.38	2.50	2.38	
			Young white wine [w]	0.05	< 0.01	0.06	0.36	0.38	
			Stored white wine, 6 months [w]	0.05	< 0.01	0.06	0.36	0.38	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	0.16/0.14 (0.15)	0.03/0.03 (0.03)	0.19/0.17 (0.18)			
			Berries for dipping	0.10	0.02	0.12	0.67	0.67	
			Dipping solution	0.02	< 0.01	0.03	0.13	0.17	
			Raisins	0.07	0.02	0.09	0.47	0.50	
81600 Tecou France (south) 2012 Merlot, (red variety)	0.1077 0.1055 (13)	29	Wine grape bunches prior to juice/wine processing (RAC)	0.05/0.06 (0.06)	0.01/0.01 (0.01)	0.06/0.07 (0.07)			JSM0350 JSM0350-4 [Schäufele, M., 2013n]
			Stems [j]	0.41	0.06	0.47	6.83	6.71	
			Must [j]	0.04	< 0.01	0.05	0.67	0.71	
			Must, after heating [j]	0.06	0.02	0.08	1.00	1.14	
			Wet pomace [j]	0.07	0.02	0.09	1.17	1.29	
			Dry pomace [j]	0.05	0.02	0.07	0.83	1.00	
			Raw juice [j]	0.05/0.08 (0.07)[c]	< 0.01/0.02 (0.02)[c]	0.06/0.10 (0.08)	1.17	1.14	
			Dregs [j]	0.18	0.07	0.25	3.00	3.57	
			Clear juice [j]	0.04/0.05 (0.05)[c]	< 0.01/0.01 (0.01)[c]	0.05/0.06 (0.06)	0.83	0.86	
			Grape juice, pasteurised [j]	0.04	< 0.01	0.05	0.67	0.71	
			Stems [w]	0.48	0.07	0.55	8.00	7.86	
			Must/Crushed grapes [w]	0.05	0.01	0.06	0.83	0.86	
			Wet pomace [w]	0.26/0.29 (0.28)[c]	0.07/0.08 (0.08)[c]	0.33/0.38 (0.36)	4.67	5.14	
			Dry pomace [w]	0.15/0.19 (0.17)[c]	0.04/0.05 (0.05)[c]	0.19/0.24 (0.22)	2.83	3.14	
			AF wine [w]	0.02	< 0.01	0.03	0.33	0.43	
			MLF wine [w]	0.01	< 0.01	0.01	0.17	0.14	
			Lees [w]	0.04	< 0.01	0.05	0.67	0.71	
			Young red wine [w]	0.01	< 0.01	0.01	0.17	0.14	
			Stored red wine, 6 months [w]	0.01	< 0.01	0.01	0.17	0.14	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	0.05/0.06 (0.06)	0.01/0.02 (0.02)	0.06/0.08 (0.07)			
			Berries for dipping	0.04	0.02	0.06	0.67	0.86	
			Dipping solution	0.01	< 0.01	0.01	0.17	0.14	

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
			Raisins	0.01	< 0.01	0.01	0.17	0.14	

AF= alcoholic fermentation; MLF maleic fermentation

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

[c] Retain specimens of raw juice, clear juice, wet and dry pomace have been analysed. The retain specimens confirmed the initial residue levels found in the “ship” specimens. Values in brackets are the mean values of “ship” and retain specimens.

[j] from juice process

[w] from wine process

Table 109 Residues of cyclaniliprole and NK-1375 after processing (follow-up study) of grapes

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
79235 Vogtsburg-Oberbergen Germany 2012 Weiss Burgunder (white variety)	0.0364 0.0323 (14)	27	Wine grape bunches prior to juice/wine processing (RAC)	0.03/0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.04/0.05 (0.05)			JSM0350 JSM0350-1 [Schäufele, M., 2013n]
			Pasteurised juice	0.01	< 0.01	0.01	0.25	0.20	
			Wet pomace	0.04	< 0.01	0.05	1.00	1.00	
			Young wine	0.02	< 0.01	0.03	0.50	0.60	
			Stored wine, 6 months	0.01	< 0.01	0.01	0.25	0.20	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	0.04/0.02 (0.03)	< 0.01/< 0.01 (< 0.01)	0.05/0.03 (0.04)			
			Raisins	0.02	< 0.01	0.03	0.67	0.75	
54200 Bruley France, 2012 Auxerrois (white variety)	0.0367 0.0391 (14)	29	Wine grape bunches prior to juice/wine processing (RAC)	0.06/0.04 (0.05)	< 0.01/< 0.01 (< 0.01)	0.07/0.05 (0.06)			JSM0350 JSM0350-2 [Schäufele, M., 2013n]
			Pasteurised juice	0.02	< 0.01	0.03	0.40	0.50	
			Wet pomace	0.06	0.03	0.09	1.20	1.50	
			Young wine	0.02	< 0.01	0.03	0.40	0.50	
			Stored wine, 6 months	0.02	< 0.01	0.03	0.40	0.50	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	0.03/0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.04/0.05 (0.05)			
			Raisins	< 0.01	< 0.01	< 0.02	0.25	< 0.40	
15050 Casaco Italy 2012 Timorasso (white	0.0359 0.0365 (15)	23	Wine grape bunches prior to juice/wine processing (RAC)	< 0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)			JSM0350 JSM0350-5 [Schäufele, M., 2013n]
			Pasteurised juice	< 0.01	< 0.01	< 0.01	[c]	[c]	
			Wet pomace	0.03	< 0.01	0.04	[c]	[c]	

Location, Country; year; (variety) variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
			Young wine	< 0.01	< 0.01	< 0.01	[c]	[c]	
			Stored wine, 6 months	< 0.01	< 0.01	< 0.01	[c]	[c]	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)			
			Raisins	< 0.01	< 0.01	< 0.02	[c]	[c]	
67117 Furdenheim France, north 2012 Pinot noir (red variety)	0.0367 0.0391 (14)	27	Wine grape bunches prior to juice/wine processing (RAC)	0.02/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.03/0.03 (0.03)			JSM0350 JSM0350-3 [Schäufele, M., 2013n] follow-up
			Pasteurised juice	0.01	< 0.01	0.01	0.50	0.33	
			Wet pomace	0.16	0.04	0.20	8.00	6.67	
			Young wine	< 0.01	< 0.01	< 0.01	< 0.50	< 0.33	
			Stored wine, 6 months	< 0.01	< 0.01	< 0.01	0.50	< 0.33	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	0.03/0.03 (0.03)	< 0.01/< 0.01 (< 0.01)	0.04/0.04 (0.04)			
			Raisins	0.02	< 0.01	0.03	0.67	0.75	
81600 Tecou France (south) 2012 Merlot, (red variety)	0.0362 0.0356 (13)	28	Wine grape bunches prior to juice/wine processing (RAC)	0.02/0.04 (0.03)	< 0.01/< 0.01 (< 0.01)	0.03/0.05 (0.04)			JSM0350 JSM0350-4 [Schäufele, M., 2013n] follow-up
			Pasteurised juice	0.01	< 0.01	0.01	0.33	0.12	
			Wet pomace	0.09	0.02	0.11	3.00	2.75	
			Young wine	< 0.01	< 0.01	< 0.01	< 0.33	< 0.25	
			Stored wine, 6 months	< 0.01	< 0.01	< 0.01	< 0.33	< 0.25	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	0.05/0.07 (0.06)	0.01/0.01 (0.01)	0.07			
			Raisins	< 0.01	< 0.01	< 0.01	< 0.17	< 0.14	
17780 Carriguella Spain 2012 Carinyena (red variety)	0.0346 0.0345 (14)	28	Wine grape bunches prior to juice/wine processing (RAC)	< 0.01/< 0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (0.01)			JSM0350 JSM0350-6 [Schäufele, M., 2013n] follow-up
			Pasteurised juice	< 0.01	< 0.01	< 0.01	[c]	[c]	
			Wet pomace	0.04	< 0.01	0.05	[c]	[c]	
			Young wine	< 0.01	< 0.01	< 0.01	[c]	[c]	
			Stored wine, 6 months	< 0.01	< 0.01	< 0.01	[c]	[c]	
idem	idem	idem	Wine grape bunches prior to processing raisins (RAC)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)			
			Raisins	< 0.01	< 0.01	< 0.01	[c]	[c]	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

[c] Since the RAC contained residues <LOQ, no reliable PF can be calculated.

Grapes, study 2

The purpose of this study was to quantify the magnitude of BPQO, BCPBA and YT-1327 (degradation products of cyclaniliprole) in wine grapes and their processed commodities [Miller, C., 2016; report BS38WY]. Samples from a former grape processing study JSM0350 [Schäufele, 2013n, report JSM0350] were analysed for BPQO, BCPBA and YT-1327.

The samples were stored at <-18 °C for 3.5 years before analyses.

BPQO, BCPA and YT-1327 were analysed using LC-MS/MS method XR44SB with overall mean recovery for BPQO, BCPBA and YT-1327 of 86%, 101% and 87%, respectively. Untreated control samples of RAC and processed commodities showed residues below the LOQ of 0.01 mg/kg.

BPQO, BPCBA and YT-1327 were not found above the LOQ of 0.01 mg/kg in any of the processed commodities specimens from any of the trials.

The storage stability of the analytes over the period of 3.5 years was not covered with storage stability studies.

Tomatoes, study 2

A processing study with tomato was conducted in 2012 at 4 sites (Italy, France (South) and The Netherlands) [Alé, 2013p, JSM0354]. The results for the part of the study, in which residues of cyclaniliprole and NK-1375 in RAC have been investigated, have been evaluated in the section with supervised residue trials.

Cyclaniliprole was applied 2 times at either nominal application rates of 0.04 kg ai/ha (four sites) or at exaggerated nominal application rates of 0.12 kg ai/ha (one site), and a 6–7 day interval to indoor tomatoes. An oilseed rape oil adjuvant (Codacide) was also added to the spray mixture. Tomatoes were harvested 1 day after the last application and were processed to canned tomatoes, juice, puree and dried tomatoes.

Canned tomatoes (the weights in brackets are for the treated fruits)

Tomatoes (1566–3147 g) were peeled and introduced into glass preserve jars (sample of 403–617 g). The jars were filled up with water (310–451 g), closed and pasteurised at 95 °C for approximately 5 minutes.

Juice (the weights in brackets are for the treated fruits)

Tomatoes (3010–4122 g; 5260 g from study 4 for both juice and puree; 8094 g from the balance study for both juice and puree) were roughly cut and passed through a vegetable mill to separate the juice from peels and seeds. The weight of the wet pomace (which includes peels and seeds) was noted (420–739 g; 783 g from study 4 for both juice and puree; 1612–8094 g from the balance study for both juice and puree). The Brix degree of raw juice was measured, the acidity of the juice was adjusted to a pH below 4.25 with citric acid. The raw juice (2362–3118 g; 4439 g from study 4 for both juice and puree; 5847 g from the balance study for both juice and puree) was heated at 95 °C for approximately 5 minutes for pasteurisation. No sub-fractionation took place.

Puree (the weights in brackets are for the treated fruits)

Tomatoes (3064–3860 g) were first processed into juice (2330–3151 g) as described above. The juice was then concentrated under vacuum and gentle heating (55–60 °C) until the desired concentration was obtained (12° Brix). After concentration, the puree (840–1010 g) was canned and heated shortly at 95 °C for 5 minutes for pasteurisation. No sub-fractionation took place.

Dried tomatoes (the weights in brackets are for the treated fruits)

Tomatoes (1422–2079 g) were cut into two parts. Prepared tomatoes were placed in a regulated ventilated dryer at 40 °C during 24–48 hours until reaching the desired water content (around 10% moisture) (weight dried tomatoes: 222–505 g). No sub-fractionation took place.

Peeled fruits and peels (only for balance study; the weights in brackets are for the treated fruits)

Tomatoes (2000 g) were peeled by submerging into boiling water for approximately 60 seconds and then place immediately into cold water for 20 seconds to crack the skin (1912 g peeled tomatoes; 75 g peel). No sub-fractionation took place.

All processed commodities were stored frozen (target temperature of -18 °C) from the day of collection onwards for a maximum of 106 days, except for one retain sample which was stored for 153 days.

Samples were analysed for cyclaniliprole and NK-1375 by LC-MS/MS (JSM0269). The LOQ for both parent and metabolite was 0.01 mg/kg. Procedural recoveries for both analytes were within the acceptable range of 70–120%. Procedural recoveries were only conducted with the RAC. No residues of cyclaniliprole and its metabolite NK-1375 were detected above the LOQ in any of the untreated RAC specimens from any of the trials.

The results are presented in Table 110.

Tomatoes, study 3

A processing study with tomatoes was conducted in 2013 in USA [Wiedmann, J.L. and McDonald, J.A. 2014c, report IB-2012-JLW-029-01-01].

An SL formulation of Cyclaniliprole was applied 3 times at exaggerated application rates of 0.604–0.607 kg ai/ha and a 6–7-day interval to tomatoes. Tomatoes were harvested 1 day after the last application. RAC samples were stored frozen from the sampling date to the shipment date. Tomato samples for processing were transported, the same day, to the processing laboratory. Tomatoes were processed to purée and paste simulating commercial practices as closely as possible.

Tomato juice

The fruit (58.42 kg) was batch soaked in a steam jacketed stainless steel kettle with ~91 kg of water and 454 grams of sodium hydroxide at 52–60 °C for 3 minutes. The fruit was batch rinsed using a high-pressure spray warm water rinse at 68–74 °C for 30 seconds per batch. The fruit was hand fed into the hammer mill assembly for crushing. The crushed tomatoes were transferred to the Steam Jacketed Kettle and rapidly heated to 79–85 °C and held for 30 seconds. The hot break juice was hand fed into the Pulper Finisher for the separation of pomace and juice. The wet pomace recovered was pressed. The recovered press juice was weighed and returned to the finished juice. Weight of juice available for puree and paste processing: 30.80 kg, a yield of 53% (30.80/58.42 kg).

Tomato puree

A subsample of 9.72 kg of the tomato juice for tomato purée was then transferred to the Laboratory 7.5 L Vacuum Evaporator by continuous feeding. The purée was removed from the evaporator when the desired Brix range was achieved. The water removed during the evaporation process was weighed and discarded. 1.0% salt and distilled water were added to adjust the Brix to the desired range of 12.0–13.0°. The purée was heated to 82–88 °C. Weight of purée after heating: 3.35 kg (10.62 kg corrected for sub fractionation). The yield from RAC to puree was 18% (3.35 kg/9.72 kg×0.53 (yield from RAC to tomato juice)). The heated purée was packed in #303 cans and sealed using the Dixie Electric Can Sealer. The sealed cans were then processed in a boiling water bath for ~15 minutes at 96–100 °C and then cooled under running cold tap water.

Tomato paste

A subsample of 21.32 kg of the tomato juice for tomato paste was transferred to the Laboratory 7.5 L Vacuum Evaporator by continuous feeding. Tomato juice in excess was weighed and discarded. The paste was removed from the evaporator when the desired Brix range was achieved. The water removed during the evaporation process was weighed and discarded. 0.5% salt and distilled water (if necessary) was added to adjust the Brix to the desired range of 24–33 °C. The remaining paste was heated to 82–88 °C. Weight of paste after heating: 4.25 kg (6.14 kg corrected for sub-fractionation). The yield from RAC to paste was 11% (4.25/21.32 ×0.53 (yield from RAC to tomato juice)). The heated paste was packed in #303 cans and sealed using the Dixie Electric Can Sealer. The sealed cans were then processed in a boiling water bath for ~15 minutes at 96–100 °C and then cooled in cold tap water.

Upon receipt at the analytical laboratory, samples were immediately inventoried and placed into freezers set to maintain < -10 °C. Freezer temperatures ranged from -42 °C to -4 °C throughout the sample storage period. Temperatures briefly exceeded -10 °C on three occasions due to equipment malfunction and/or maintenance and once during receipt of a large shipment of samples; however, during these excursions, the samples remained frozen. The maximum storage duration for tomato samples (RAC and processed samples) was 71 days.

Samples were analysed for parent cyclaniliprole and metabolite NK-1375 using LC-MS/MS analytical method JSM0269.

Laboratory fortification samples were analysed concurrently with each analytical set. The laboratory fortification samples were prepared using untreated samples obtained from a field site, except for tomato puree and paste, which were prepared using controls purchased from a commercial source. The concurrent laboratory fortification recoveries demonstrated that the method worked well throughout the study. All laboratory fortification sample recoveries were within the acceptable range of 70% to 120%.

Cyclaniliprole or NK-1375 residues were below the LOQ of 0.01 mg/kg in any of the untreated field samples and untreated processed samples (puree and paste).

The results are presented in Table 110.

Table 110 Residues of cyclaniliprole and NK-1375 in tomatoes after processing

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
Wellerllooi, the Netherlands, 2012 (270-202RZ)	0.0404 0.0394 (7)	1	Fruits	0.05/0.05 (0.05)	< 0.01/< 0.01 (< 0.01)	0.06			JSM0354 JSM0354-01 [Alé E., 2013p] see section residue trials
			Canned tomato	< 0.01	< 0.01	< 0.01	< 0.20	< 0.16	
			Juice	0.01	< 0.01	0.01	0.20	0.16	
			Puree [c]	0.03	< 0.01	0.04	0.60	0.67	
			Dried tomatoes	0.19	< 0.01	0.20	3.8	3.33	
Fronton, France, 2012 (Corazon)	0.0435 0.0423 (7)	1	Fruits	0.01/0.01 (0.01)	< 0.01/< 0.01 (< 0.01)	0.01			JSM0354 JSM0354-02 [Alé E., 2013p] see section residue trials
			Canned tomato	< 0.01	< 0.01	< 0.01	<1	< 0.5	
			Juice	< 0.01	< 0.01	< 0.01	<1	< 0.5	
			Puree [c]	0.01	< 0.01	0.01	1	0.5	
			Dried tomatoes	0.04	< 0.01	0.05	4	5	
Baldisero D'Álba, Italy, 2012 (Rugantino)	0.0410 0.0444 (6)	1	Fruits	0.04/0.04/0.04 (0.04)	< 0.01/< 0.01 (< 0.01)	0.05			JSM0354 JSM0354-03 [Alé E., 2013p] see section residue trials
			Canned tomato	< 0.01/< 0.01 (< 0.01)	< 0.01/< 0.01 (< 0.01)	< 0.01	< 0.25	< 0.2	
			Juice	0.03	< 0.01	0.04	0.75	0.8	
			Puree [c]	0.08	< 0.01	0.09	2	1.8	
			Dried tomatoes	0.17/0.12	< 0.01/< 0.01	0.18/0.13	3.75	3.2	

Location, Country; year; (variety)	Treatment (kg ai/ha, (interval))	DALT	Processed products	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
				(0.15)	(< 0.01)	(0.16)			
Bra, Italy, 2012 (Ingrid)	0.0405 0.0407 (7)	1	Fruits	0.01/0.02 (0.02)	< 0.01/< 0.01 (< 0.01)	0.02/0.03 (0.03)			JSM0354 JSM0354-04 [Alé E., 2013p] see section residue trials
			Canned tomato	< 0.01	< 0.01	< 0.01	< 0.5	< 0.5	
			Juice	0.02	< 0.01	0.03	1	1.5	
			Puree [c]	0.04	< 0.01	0.05	2	2.5	
			Dried tomatoes	0.11	< 0.01	0.12	5.5	6	
	0.1219 0.1219 (7)	1	Fruits	0.06/0.05 (0.06)	< 0.01/< 0.01 (< 0.01)	0.07/006 (0.07)			JSM0354 JSM0354-04 [Alé E., 2013p] see section residue trials
			Washed tomatoes	0.04	< 0.01	0.05	0.67	0.71	
			Washing water	0.01	< 0.01	0.01	0.17	0.14	
			Peeled tomatoes	< 0.01	< 0.01	< 0.01	< 0.17	< 0.14	
			Peel and water	0.02	< 0.01	0.03	0.33	0.43	
			Canned tomatoes	< 0.01	< 0.01	< 0.01	< 0.17	< 0.14	
			Wet pomace	0.04	< 0.01	0.05	0.67	0.71	
			Dry pomace	1.32	< 0.01	1.33	22	19	
			Raw juice	< 0.01	< 0.01	< 0.01	< 0.17	< 0.14	
			Juice	0.07	< 0.01	0.08	1.17	1.14	
			Puree[c]	0.10/0.09 (0.10)	< 0.01	0.11	1.67	1.57	
			Dried tomatoes	0.20	< 0.01	0.21	3.33	3	
Porterville, CA 93257 USA, 2013 (Variety: AB2)	0.604 0.607 0.606 (6-7)	1	Fruits	0.149/ 0.130/ 0.214 (0.164)	0.014/ 0.018/ 0.020 (0.017)	0.164/ 0.150/ 0.235 (0.183)			IB-2012-JLW-029-01-01 [Wiedmann, J.L. and McDonald, J.A. 2014c]
			Puree	0.037/ 0.038 (0.038)	< 0.01/ < 0.01 (< 0.01)	0.047 0.049 (0.048)	0.23	0.26	
			Paste	0.071/ 0.080 (0.076)	0.012/ 0.014 (0.013)	0.084/ 0.094 (0.089)	0.46	0.49	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF parent = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) / residue of parent cyclaniliprole + NK-1375 in raw agricultural commodity (mg/kg).

[c] Since the yield from RAC to puree was 18%, the PFs for puree from these trials can be used to establish a median PF for tomato paste, instead of puree.

Tea, study 1 and 2

Two processing studies with tea were conducted in Japan in 2011 and 2012 [Koki, 2012, report JP2011C133 and Yosiyuki, 2013, report JP2012C101]. The purpose of these studies was to quantify the magnitude and decline of residues of cyclaniliprole and its metabolite NK-1375 in tea infusions. An SL formulation of cyclaniliprole containing 50 g ai/kg was applied once at application rates of 171–199 g ai/ha to tea trees. Details of the studies are summarised in the section of field residue trials.

Tea leaves (3–5 leaf stage) were harvested 3 days after application. The fresh tea leaves were dried to crude tea by vapour heating at 100 °C for 60 seconds or through belt conveyor (speed 1 m/45 seconds, 40 kg/h, 45 seconds or 60 kg/h, 35 seconds). The steamed leaves were dried at 80 °C for about 90–140 minutes) on the day of sampling to produce crude tea.

Hot water infusion

The crude leaves were processed to hot water infusion by adding 360 mL hot water to 6 g crude tea. The mixture was allowed to stand for 5 minutes.

Samples were analysed for cyclaniliprole and NK-1375 using LC-MS/MS Method JP2011C133 (see analytical section). The storage period ranged from 99–167 days [Yoshiyuki, 2013f, report JP2012C101 and Koki, 2012, report JP2011C133] and stability was covered in the same studies.

The residues of cyclaniliprole and the processing factors for the hot water infusion are summarised in Table 111.

Table 111 Summary of cyclaniliprole residues and processing factors for cyclaniliprole in tea

Location, Country; year; (variety)	Treatment (g ai/ha)	Processed products	DALT	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
Naruto, Sanmu-shi Chiba, Japan, 2011 (Yabukita)	199	RAC	3	8.41/8.35 (8.38)	2.13/2.08 (2.10)	10.7/10.6 (10.6)	-	-	JPPA-Chiba, JP201C133A [Koki M, 2012]
		hot water infusion	3	1.67 / 1.61 (1.64)	0.12 / 0.11 (0.12)	1.80/1.72 (1.76)	0.20	0.17	
		RAC	7	3.14 / 3.11 (3.13)	0.55 / 0.54 (0.54)	3.73/3.68 (3.70)	-	-	
		hot water infusion	7	0.67 / 0.65 (0.66)	0.04 / 0.03 (0.04)	0.71/0.68 (0.70)	0.21	0.19	
		RAC	14	0.36 / 0.35 (0.36)	0.12 / 0.12 (0.12)	0.49/0.48 (0.48)	-	-	
		hot water infusion	14	0.06/0.06 (0.06)	< 0.02 / < 0.02 (< 0.02)	0.08/0.08 (0.08)	0.17	0.17	
		RAC	21	< 0.02/< 0.02 (< 0.02)	< 0.02 / < 0.02 (< 0.02)	< 0.04/< 0.04 (< 0.04)	-	-	
		hot water infusion	21	< 0.02 / < 0.02 (< 0.02)	< 0.02 / < 0.02 (< 0.02)	< 0.04/< 0.04 (< 0.04)	-	-	
Niyodogawa-cho Mori, Agawa-gum Kochi, Japan, 2011 (Yabukita)	191	RAC	3	4.88 / 4.78 (4.83)	0.09 / 0.09 (0.09)	4.98/4.88 (4.93)	-	-	JPPA-Kochi, JP201C133B [Koki M, 2012]
		hot water infusion	3	0.61/0.60 (0.61)	< 0.02 / < 0.02 (< 0.02)	0.63/0.62 (0.63)	0.13	0.13	
		RAC	7	3.18 / 3.03 (3.10)	0.11 / 0.11 (0.11)	3.29/3.15 (3.22)	-	-	
		hot water infusion	7	0.29 / 0.25 (0.27)	< 0.02 / < 0.02 (< 0.02)	0.31/0.27 (0.29)	0.09	0.09	
		RAC	14	0.46 / 0.45 (0.46)	0.31 / 0.30 (0.30)	0.79/0.77 (0.78)	-	-	
		hot water infusion	14	0.05 / 0.05 (0.05)	< 0.02 / < 0.02 (< 0.02)	0.07/0.07 (0.07)	0.11	0.09	
		RAC	21	< 0.02 / < 0.02 (< 0.02)	< 0.02 / < 0.02 (< 0.02)	< 0.02/< 0.02 (< 0.02)	-	-	
		hot water infusion	21	< 0.02 / < 0.02 (< 0.02)	< 0.02 / < 0.02 (< 0.02)	< 0.02/< 0.02 (< 0.02)	-	-	
Kamiyaganuki, Iiruma-shi, Saitama, Japan, 2012 (Sayama-midori)	185	RAC	3	13.0 / 12.9 (13.0)	1.31 / 1.30 (1.31)	14.4/14.3 (14.3)	-	-	Saitama-Tea JP2012C101 A [Yoshiyuki, 2013f]
		hot water infusion	3	1.83 / 1.70 (1.77)	0.05 / 0.05 (0.05)	1.88/1.75 (1.82)	0.14	0.13	
Naruto, Sanmu-shi Chiba, Japan, 2012 (Yabukita)	174	RAC	3	6.84 / 6.66 (6.75)	0.68 / 0.66 (0.67)	7.56/7.63 (7.46)	-	-	JPPA-Chiba JP2012C101B [Yoshiyuki, 2013f]
		hot water infusion	3	1.27 / 1.22 (1.24)	0.03 / 0.03 (0.03)	1.30/1.25 (1.28)	0.18	0.17	

Location, Country; year; (variety)	Treatment (g ai/ha)	Processed products	DALT	Residues, mg/kg			PF[a] parent	PF[b] Tot.	Report; Trial no [ref]
				Parent	NK-1375	Tot.			
Niyodogawa-cho Mori, Agawa-gum Kochi, 2012 (Yakubita)	189	RAC	3	28.3 / 27.8 (28.0)	1.46 / 1.36 (1.41)	29.9/29.2 (29.6)	-		JPPA-Kochi JP2012C101C [Yoshiyuki, 2013f]
		hot water infusion	3	2.72 / 2.67 (2.70)	0.04 / 0.04 (0.04)	2.76/2.72 (2.74)	0.10	0.09	
Nagasato, Chiran-cho, Minamikyusyu-shi Kagoshima, 2012 (Yamato-midori)	171	RAC	3	16.5 / 16.2 (16.4)	0.62 / 0.62 (0.62)	17.2/16.9 (17.0)	-	-	Kagoshima-Tea JP2012C101D [Yoshiyuki, 2013f]
		hot water infusion	3	2.46 / 2.34 (2.40)	0.03 / 0.03 (0.03)	2.49/2.37 (2.43)	0.15	0.14	

[a] PF parent = residue of parent cyclaniliprole in processed product (mg/kg) / residue of parent cyclaniliprole in raw agricultural commodity (mg/kg).

[b] PF total = residue of parent cyclaniliprole + NK-1375 in processed product (mg/kg) x 1.064, expressed as cyclaniliprole / residue of parent cyclaniliprole + NK-1375, expressed as cyclaniliprole in raw agricultural commodity (mg/kg).

Residues in tea before processing were < 0.02 to 28.1 mg/kg cyclaniliprole and < 0.02 to 2.11 mg/kg NK-1375. Mean residues in hot water infusions were in the range between < 0.02 and 2.70 mg/kg cyclaniliprole and < 0.02 to 0.12 mg/kg for NK-1375. The resulting median processing factor for cyclaniliprole was 0.14 for hot water infusions. The median processing factor for total residues, including NK-1375 was 0.14 as well. No concentration of residues of cyclaniliprole occurs.

Residues in the edible portion of food commodities

No studies were performed in which residue were measured in the RAC as well as the edible portion. However, several studies on stone fruit include data on the edible portion of the fruit only, without providing data on the different fractions of the fruit. As the RAC includes the stone, but the stone is not expected to hold residue, a dilution of the residue to the RAC is expected. In some trials the weights of fruit, flesh and stone were reported. This data is summarized for use with data where this information is lacking.

Field residue trials on cherries were carried out in Japan [Kuzaki and Naruto, 2013]. Residues were determined in flesh (de-pitted fruit). The report also contains data on the ratio of whole fruit, flesh, and seed. The results are summarised in Table 112. The ratio of fruit to whole fruit was calculated and used to convert the residue values reported on flesh to the RAC (whole fruit). The average and median ratio is 1.14 and 1.13, respectively.

Table 112 Weight ratios for whole fruit, flesh and seed of cherries from field residue trials

Location	DAT	mean weight (gram/piece)	mean weight (g/piece)	mean weight (g/piece)	Ratio whole fruit/fruit	Recovery (%)[a]	Reference
		Whole fruit	Fruit	Seed			Kuzaki and Naruto, 2013, report no code
Fukushima, Japan	0	7.41	6.39	0.47	1.16	93	
	1	8.44	7.56	0.55	1.12	96	
	3	8.42	7.53	0.54	1.12	96	
	7	7.84	7.03	0.49	1.12	96	
	14	7.91	6.99	0.53	1.13	95	
	21	7.73	7.04	0.50	1.10	98	
Suzaka, Japan	0	5.84	5.09	0.57	1.15	97	
	1	6.50	5.76	0.51	1.13	96	
	3	6.02	5.24	0.49	1.15	95	
	7	6.25	5.49	0.52	1.14	96	

Location	DAT	mean weight (gram/piece)	mean weight (g/piece)	mean weight (g/piece)	Ratio whole fruit/fruit	Recovery (%)[a]	Reference
	14	5.65	4.71	0.53	1.20	93	
	21	5.92	5.22	0.52	1.13	97	
				mean	1.14		
				median	1.13		

[a] Calculated as ((sum of fruit and seed) /total mean weight) * 100. It is noted that the total mean weights are generally higher than the calculated total weights used to determine the ratio.

Table 113 presents an overview of processing factors which were considered acceptable for refinement of the dietary risk assessment and/or dietary burden calculations. Processing factors are calculated both for parent only and for parent NK-1375 by dividing the residue concentrations in the processed commodity by the respective residue concentrations in the RAC.

Table 113 Overview of processing factors of incurred processing studies

Commodities	PF (parent only) individual acceptable results	PF (parent only) Median, mean or best estimate (BE)	PF (parent+ NK-1375) individual acceptable results	PF (parent+NK1375) Median, mean or best estimate
apples				
- canned fruit	< 0.5, < 0.5	< 0.5 (mean, n=2)	< 0.33, < 0.33	< 0.33 (mean, n=2)
- purée	< 0.5, < 0.5	< 0.5 (mean, n=2)	< 0.33, < 0.33	< 0.33 (mean, n=2)
- juice, pasteurised	< 0.5, < 0.5, 0.13	0.13 (BE, n=3)	< 0.33, < 0.33, 0.13	0.13 (BE n=3)
- wet pomace	3.2	3.2 (n=1)	3.2	3.2 (n=1)
peaches				
- canned	< 0.14, < 0.5, < 0.5, < 0.5, <1	< 0.5 (median, n=5)	< 0.1, < 0.33, < 0.33, < 0.33, < 0.5	< 0.33 (median, n=5)
- juice/nectar, pasteurised	< 0.14, < 0.5, < 0.5, < 0.5, <1	< 0.5 (median, n=5)	< 0.1, < 0.33, < 0.33, < 0.33, < 0.5	< 0.33 (median, n=5)
- purée, after pasteurisation	< 0.14, < 0.5, < 0.5, < 0.5, <1	< 0.5 (median, n=5)	< 0.1, < 0.33, < 0.33, < 0.33, < 0.5	0.33 (median, n=5)
plums				
- dried prunes	3.7	3.7 (n=1)	3.7	3.7 (n=1)
grapes				
- must	0.64[j], 0.64[w], 0.67[j], 0.83[w]	0.66 (median n=4)	0.63[j], 0.63[w], 0.71[j], 0.86[w]	0.67 (median, n=4)
- must, after heating	1.0	1.0 (n=1)	1.14	1.1 (n=1)
- wet pomace	1.0[f], 1.17[j], 1.20[f], 1.64[j], 1.71[w], 3.0[f], 4.67[w], 8.0[f],	1.68 (median, n=8)	1.0[f], 1.29[j], 1.50[f], 1.63[j], 1.75[w], 2.75[f], 5.14[w], 6.67[f]	1.69 (median, n=8)
- dry pomace	0.83[j], 3.07[j], 3.07[w], 2.83[w]	3.07 (median, n=4)	1.0[j], 3.13[j], 3.13[w], 3.14[w]	3.13 (median, n=4)
- clear juice	0.43[j], 0.83[j]	0.63 (mean, n=2)	0.44 [j], 0.86[j]	0.65 (mean, n=2)
- juice after pasteurisation	0.25[f], 0.33[f], 0.36[j], 0.40[f], 0.50[f], 0.67[j]	(median, n=6)	0.20[f], 0.12[f], 0.33[f], 0.38[j], < 0.25[f], 0.71[j]	0.36 (median, n=6)
- young wine	0.17, < 0.33[f], 0.36, 0.40 [f], < 0.50[f], 0.50[f]	(median, n=6)	0.14, < 0.25[f], 0.28, < 0.33[f], 0.38, 0.50[f], 0.60[f]	(median, n=6)
- stored wine	0.17, 0.25[f], < 0.33[f], 0.36, 0.40[f], 0.50[f]	(median, n=6)	0.14, 0.20[f], 0.20[f], < 0.25[f], 0.38, 0.50[f]	0.355 (median, n=6)
- raisins	< 0.17[f], 0.17, < 0.25[f], 0.47, 0.67[f], 0.67[f]	0.47 (median, n=5)	< 0.14[f], 0.14, < 0.20[f], 0.50, 0.75 [f], 0.75[f]	0.5 (median, n=5)
tomatoes				
- canned	< 0.17, 0.20, < 0.25, < 0.5, <1	< 0.25 (median, n=5)	< 0.14, < 0.17, < 0.2, < 0.5, < 0.5	< 0.2 (median, n=5)
- wet pomace	0.67	0.67 (n=1)	0.71	0.71 (n=1)
- dry pomace	22	22 (n=1)	19	19 (n=1)
- purée (USA)	0.23	0.23 (n=1)	0.26	0.26 (n=1)
- paste USA + purée (EU) [u]	0.46[u], 0.60, 1, 1.67, 2, 2	1.34 (median n=6)	0.49[u], 0.5, 0.67, 1.57, 1.8, 2.5	1.12 (median n=6)
- juice, pasteurised	0.20, 0.75, <1, 1, 1.17	1 (median n=5)	< 0.5, 0.17, 0.8, 1.14, 1.5	0.8 (median n=5)

Commodities	PF (parent only) individual acceptable results	PF (parent only) Median, mean or best estimate (BE)	PF (parent+ NK-1375) individual acceptable results	PF (parent+NK1375) Median, mean or best estimate
- dried tomatoes	3.33, 3.75, 3.8, 4, 5.5	3.8 (median n=5)	3, 3.2, 3.3, 5, 6	3.3 (median n=5)
tea				
- hot water infusion	0.10, 0.11[t], 0.14, 0.15, 0.18, 0.19[t]	0.145 (median n=6)	0.09, 0.10[t], 0.13, 0.14, 0.17, 0.18[t]	0.135 (median n=6)

[j] From juice process and [w] from wine process, but both within the same study;

[f] From follow up study.

[t] Mean of triplicate infusions (DALT 3, 7 and 14 days) from one site

[u] Values include PFs derived in European studies defining the processed product as puree. Since evaporation was used to concentrate the volume about 3 times, the European data for purée were scaled under paste. The value assigned [u] was the USA trial.

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Not applicable

Farm animal feeding studies

The Meeting received information on feeding studies in dairy cows.

A residue feeding study in lactating Holstein/Friesian dairy cows (9-12 years old) was conducted in the USA in 2012 [Ross V., 2013, report JSM0515] to quantify the residues of cyclaniliprole and its significant metabolites, NK-1375, NSY-27, NSY-28 and YT-1284 found in milk and tissues. Cyclaniliprole was administered orally in gelatine capsules twice daily to three - six cows per dose group for 28-31 consecutive days. The cows received 0, 5, 15, or 50 mg a.i./650 kg animal/day. Three animals were maintained in the high dose group for up to 2 weeks after cessation of treatment in order to provide data on the decline of any incurred residues. Experimental conditions are indicated in Table 114. Animals were observed several times daily for any clinical signs of toxicity or ill health. Bodyweights were determined at intervals and concentrate food/hay consumption was monitored daily. Milk yields were recorded twice daily. Milk samples were taken from all cows daily throughout the test period and were submitted for assay or stored frozen. At termination of the experimental period, all cows were sacrificed and examined macroscopically post mortem. Selected tissues were retained for determination of residue concentrations.

The cows weighed 532–694 kg at the start of treatment and had a milk yield of 12.3–23.4 L/day.

Milk samples were collected daily throughout acclimation and feeding periods (days 7, and 1 to termination of the study). On days 21 and 28 additional samples of whole milk were collected for determination of fat content. Furthermore, skimmed milk and cream were produced from milk collected at days 21 and 28.

All remaining treated cows and one cow from the control group were euthanised on days 29–43. Samples of liver (whole organ as two subsamples), kidney (whole organ as two subsamples), muscle (loin/flank/hind leg pooled samples) and fat (subcutaneous, perirenal, and omental) were collected from all animals. Milk and tissue samples were stored at -20 °C until analysed.

The results from the storage stability investigations demonstrated that IKI-3106, NK-1375, NSY-27, NSY-28, and YT-1284 were stable in bovine liver, kidney, muscle and fat for a period of 39 days. All milk, skimmed milk, and cream samples were analysed within <30 days after being generated.

All study tissue samples were analysed within < 39 days after they were generated, i.e. within the period covered by the storage stability determination.

Samples were analysed for parents, NK-1375, NSY-27, NSY-28 and YT-1284 using HPLC-MS/MS method JSM0277 and using matrix-matched standards. Method JSM0277 is considered valid for the purpose of this study (commodity type and concentration level of the analytes). Residues of parents, NK-1375, NSY-27, NSY-28 and YT 1284, were not found in any of the control samples (each < 0.01 mg/kg).

Analytical results in tissue samples are shown in Table 115. Residues of parent were highest in liver. Residues in other tissues decreased in the order liver>kidney>fat>muscle for both parents. No detectable residues of NK-1375 and NSY-27 were not found in any of the tissues and were therefore not included in the table. Metabolite YT 1284 was either not detected or found only at levels below the LOQ of < 0.01 mg/kg at the higher dose levels. Residue levels of NSY-28 were only detected at the highest dose, with a maximum of 0.032 mg/kg in liver.

Table 114 Experimental conditions of the cow feeding study

Cow number	no. of days dosed	no. of depuration days	Mean group bodyweight at start and end of the feeding period (kg)	Mean daily dry feed intake (kg feed/animal)	Actual mean dose (ppm ai in feed)	Actual mean dose (mg ai/animal/day)	Actual mean dose (mg/kg bw/day)
1, 2, 3	≥28	-	613–604	3.6	0	0	0
4, 5, 6	≥28	-	628–659	4.0	1.2	4.9	0.0075
7, 8, 9	≥28	-	597–587	4.0	3.5	13.9	0.021
10, 11, 12, 13 14 15	28 28 28 28	- 2 7 14	628–642	4.2	11.6	48.8 [a]	0.075

[a] Mean of 6 cows over days 1-28 only

Total residues are calculated using “0” for non–detects and 0.01 for <LOQ + conversion factors based on molecular weight. Where both parent and the metabolites were below the LOQ the total residue was set at LOQ of 0.01 mg/kg.

Table 115 Cyclaniliprole related residues in tissues for 1.2, 3.5, and 11.6 ppm groups

Sample	Dose rate (ppm feed)	Cow	parent (mg/kg)	NSY-28 (mg/kg	YT-1284 (mg/kg)	total expressed in parent equiv. (mg/kg)[a]	mean parent (mg/kg)	max parent (mg/kg)	mean expressed in total parent eq. (mg/kg)[a]	max expressed in total parent eq. (mg/kg)[a]
Muscle	1.2	4	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01
		5	ND	ND	ND	ND				
		6	ND	ND	ND	ND				
	3.5	7	ND	ND	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01
		8	ND	ND	ND	ND				
		9	< 0.01	ND	ND	< 0.01				
	11.6	10	< 0.01	< 0.01	ND	< 0.01	0.018	0.032	0.026	0.044
		11	0.032	< 0.01	ND	0.044				
		12	0.011	< 0.01	ND	0.023				
	depuration	13	< 0.01	ND	ND	< 0.01	< 0.01	< 0.01	NA	NA
		14	< 0.01	< 0.01	ND	< 0.01				
		15	ND	ND	ND	ND				
Liver	1.2 1X dose)	4	< 0.01	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		5	< 0.01	ND	ND	< 0.01				
		6	< 0.01	< 0.01	ND	< 0.01				
	3.5	7	0.012	< 0.01	ND	0.024	0.021	0.040	0.037	0.063
		8	0.011	< 0.01	ND	0.023				
		9	0.040	< 0.01	< 0.01	0.063				
	11.6	10	0.025	0.014	< 0.01	0.053	0.071	0.141	0.11	0.19
		11	0.141	0.032	< 0.01	0.19				

Sample	Dose rate (ppm feed)	Cow	parent (mg/kg)	NSY-28 (mg/kg	YT-1284 (mg/kg)	total expressed in parent equiv. (mg/kg)[a]	mean parent (mg/kg)	max parent (mg/kg)	mean expressed in total parent eq. (mg/kg)[a]	max expressed in total parent eq. (mg/kg)[a]
		12	0.048	0.021	< 0.01	0.084				
	depuration	13	0.027	0.016	< 0.01	0.057	NA	NA	NA	NA
		14	0.023	< 0.01	ND	0.035				
		15	< 0.01	< 0.01	ND	0.022				
Kidney	1.2	4	0.011	ND	ND	0.011	< 0.01	0.011	0.010	0.011
		5	< 0.01	ND	ND	< 0.01				
		6	< 0.01	ND	ND	< 0.01				
	3.5	7	0.010	ND	ND	0.010	0.022	0.045	0.022	0.045
		8	< 0.01	ND	ND	< 0.01				
		9	0.045	ND	ND	0.045				
	11.6	10	0.020	< 0.01	< 0.01	0.043	0.059	0.114	0.079	0.14
		11	0.114	0.014	< 0.01	0.14				
		12	0.043	< 0.01	ND	0.055				
	depuration	13	0.030	< 0.01	ND	0.042	NA	NA	NA	NA
		14	0.014	ND	ND	0.014				
		15	ND	ND	ND	ND				
Subcutaneous fat	1.2	4	< 0.01	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		5	< 0.01	ND	ND	< 0.01				
		6	< 0.01	ND	ND	< 0.01				
	3.5	7	< 0.01	ND	ND	< 0.01	0.021	0.042	0.021	0.042
		8	< 0.01	ND	ND	< 0.01				
		9	0.042	ND	ND	0.042				
	11.6	10	0.015	< 0.01	< 0.01	0.038	0.061	0.199	0.083	0.15
		11	0.119	0.014	< 0.01	0.15				
		12	0.048	< 0.01	ND	0.060				
	depuration	13	0.024	< 0.01	ND	0.036	NA	NA	NA	NA
		14	0.021	ND	ND	0.021				
		15	ND	ND	ND	ND				
Perirenal fat	1.2	4	< 0.01	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		5	< 0.01	ND	ND	< 0.01				
		6	< 0.01	ND	ND	< 0.01				
	3.5	7	0.011	ND	ND	0.011	0.023	0.045	0.027	0.045
		8	0.014	< 0.01	ND	0.026				
		9	0.045	ND	ND	0.045				
	11.6	10	0.034	< 0.01	< 0.01	0.057	0.074	0.120	0.092	0.14
		11	0.120	< 0.01	< 0.01	0.14				
		12	0.067	< 0.01	ND	0.079				
	depuration	13	0.057	< 0.01	ND	0.069	NA	NA	NA	NA
		14	0.030	< 0.01	ND	0.042				
		15	ND	ND	ND	ND				
Omental fat	1.2	4	< 0.01	ND	ND	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
		5	< 0.01	ND	ND	< 0.01				
		6	< 0.01	ND	ND	< 0.01				
	3.5	7	0.011	ND	ND	0.011	0.020	0.036	0.020	0.036
		8	0.012	ND	ND	0.012				
		9	0.036	ND	ND	0.036				
	11.6	10	0.024	< 0.01	ND	0.036	0.059	0.100	0.073	0.12
		11	0.100	< 0.01	< 0.01	0.12				
		12	0.052	< 0.01	ND	0.064				
	depuration	13	0.051	< 0.01	ND	0.063	NA	NA	NA	NA
		14	0.025	< 0.01	ND	0.037				
		15	ND	ND	ND	ND				

ND=not detectable (< 0.003 mg/kg); NA=not applicable

[a] For calculation of total residue in parent equivalents the results of ND were assigned values of 0 and < LOQ and 0.01 respectively. Conversion factors (1.165 for NSY-28 and 1.128 for YT-1284) based on molecular weight difference were

used only if concentrations were at or above LOQ. Where both parent and metabolite were below LOQ the total was set at LOQ

Cyclaniliprole-derived residues above the limit of quantitation were found only in a very few whole milk samples; i.e. one animal treated at a level of 1.2 and one animal treated at 12 ppm. These levels were in the range of 0.010 to 0.016 mg/kg and only occurred on intermittent days of the dosing period (day 14, 23, 25, and 28). The level seen in the animal from Group 2 (1.2 ppm) is considered spurious as this was also seen on one occasion in a control. Residues were generally below the limit of quantitation (< 0.01 mg/kg) throughout the treatment and depuration periods. No residues of cyclaniliprole or its metabolites were detected above the limit of quantitation (0.01 mg/kg) in any samples taken on days 21 and 28 in skimmed milk. In treated animals, quantifiable residues in cream occurred only as the parent compound, and there was a correlation between treatment level and residues found: mean values were <LOQ, 0.019 and 0.078 mg/kg in Groups 2, 3, and 4 respectively at day 21 and <LOQ, 0.021 and 0.077 mg/kg at day 28. Residues in cream for the 21- and 28-day samples are shown in Table 116.

Table 116 Cyclaniliprole related residues in milk cream for 1.2, 3.5 and 11.6 dose groups

Dose rate ppm feed	Sampling	Cow	parent mg/kg
1.2	Day 21	4	0.010
		5	0.015
		6	< 0.01
	Day 28	4	0.010
		5	< 0.01
		6	< 0.01
	Average		0.011
	Highest residue		0.015
3.5	Day 21	7	0.014
		8	0.016
		9	0.028
	Day 28	7	0.015
		8	0.013
		9	0.034
	Average		0.02
	Highest residue		0.034
11.6	Day 21	10	0.069
		11	0.087
		12	0.070
		13	0.073
		14	0.077
		15	0.092
	Day 28	10	0.048
		11	0.125
		12	0.066
		13	0.045
		14	0.104
		15	0.076
	Average		0.078
	Highest residue		0.125

RESIDUES IN FOOS IN COMMERCE OR AT CONSUMPTION

Not applicable.

NATIONAL RESIDUE DEFINITIONS

Cyclaniliprole is a new substance. Applications for registration have been submitted in several countries, however, only authorisations in the Republic of Korea and the USA have been granted.

The national residue definitions there are:

Republic of Korea:

Residue definition for enforcement: *cyclaniliprole only*

Residue definition for dietary risk assessment: *cyclaniliprole only*

USA:

Residue definition for enforcement: only cyclaniliprole 3-bromo-N-[2-bromo-4-chloro-6-[[(1-cyclopropylethyl)amino]carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide

Residue definition for dietary risk assessment: not applicable

APPRAISAL

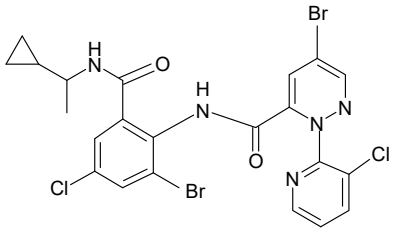
Cyclaniliprole (ISO common name) was scheduled for residue evaluation as a new compound by the 2017 JMPR at the 48th Session of the CCPR.

Cyclaniliprole is a broad-spectrum insecticide belonging to the diamide and pyrazole chemical classes of insecticides. Despite its structural similarity to some of the phenylpyrazole insecticides, this substance has a different mode of action, which it shares with other diamide insecticides. Diamides act at the ryanodine receptor, which is critical for muscle contraction.

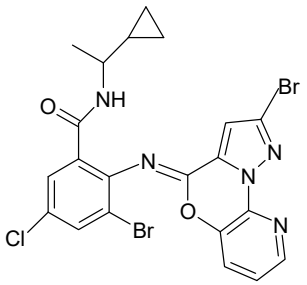
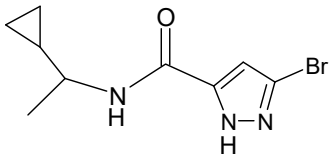
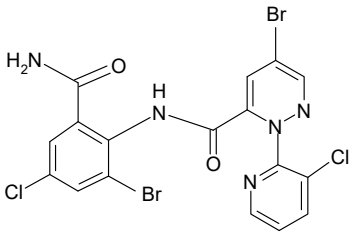
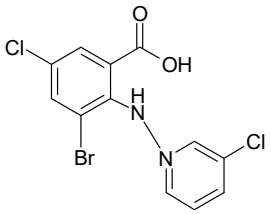
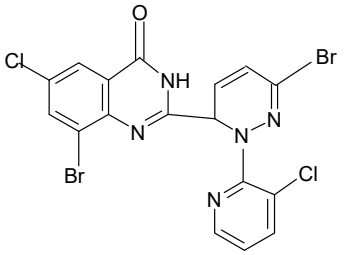
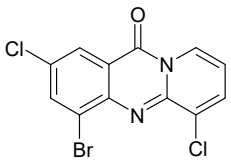
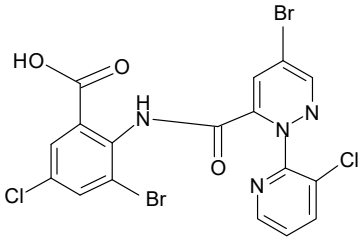
The Meeting received information from the manufacturer on identity, metabolism, environmental fate, storage stability, residue analysis, use patterns, residues resulting from supervised trials on pome fruit, stone fruit, grapes, brassica's, fruiting vegetables, leafy vegetables, soya beans, potato, almond, pecan, tea, fate of residue during processing, and livestock feeding studies.

The IUPAC name for cyclaniliprole is 2',3-dibromo-4'-chloro-1-(3-chloro-2-pyridyl)-6'-{[(1*RS*)-1-cyclopropylethyl]carbamoyl}pyrazole-5-carboxanilide. The CA name is 3-bromo-*N*-[2-bromo-4-chloro-6-[[(1-cyclopropylethyl)amino]carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide.

Structural formula



Metabolites referred to in the appraisal by codes:

NK-1375 3-bromo-2-((2-bromo-4*H*- pyrazolo[1,5-d]pyrido[3,2-b]-[1,4]oxazin-4-ylidene)amino)-5-chloro-*N*-(1-cyclopropylethyl)benzamide CF = 1.064 	YT-1327 3-bromo-*N*-(1-cyclopropylethyl)-1*H*-pyrazole-5-carboxamide 
YT-1284 3-bromo-*N*-(2-bromo-6-carbamoyl-4-chlorophenyl)-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-carboxamide CF = 1.128 	BCPBA 3-bromo-5-chloro-2-((3-chloropyridin-2-yl) amino) benzoic acid 
NSY-28 8-bromo-2-(3-bromo-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-yl)-6-chloroquinazolin-4(3*H*)-one CF = 1.165 	BPQO 4-bromo-2,6-dichloro-11*H*-pyrido[2,1-*b*]quinazolin-11-one 
NSY-27 3-bromo-2-(3-bromo-1-(3-chloropyridin-2-yl)-1*H*-pyrazole-5-carboxamido)-5-chlorobenzoic acid CF = 1.125 	

Plant metabolism

The Meeting received plant metabolism studies for cyclaniliprole after foliar application on fruits (apples), leafy vegetables (lettuce) and root and tuber vegetables (potato).

The metabolism of ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cyclaniliprole in commercially grown apples was studied following three foliar applications at 96–100 g ai/ha at a four-week interval. Total radioactive residues (TRR) in mature apples at DALA = 15 and 30 were 0.15 and 0.042 mg/kg eq for the phenyl label and 0.14 and 0.036 mg/kg eq for the pyrazole label, respectively. A high proportion of the residue remained on the surface of the fruit (59–92% TRR in surface wash; 5.3–29% TRR in peel; 2.3–12% TRR in flesh). The residues in or on the fruit could be extracted by acetonitrile/water (> 89% TRR). The principal component of the residue was the parent compound (40–50% TRR), followed by metabolite NK-1375 (23–29% TRR) and YT-1284 (1.0–3.9% TRR). A number of other metabolites were detected (0.3–4.9% TRR), but none reaching > 0.01 mg/kg eq in apple fruit.

The metabolism of ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cyclaniliprole in lettuce was studied following three foliar applications at 107–117 g ai/ha at a 10 days interval. Total radioactive residues (TRR) in mature lettuce at DALA = 8 and 15 were 0.76 and 0.39 mg/kg eq for the phenyl label and 0.76 and 0.37 mg/kg eq for the pyrazole label, respectively. A high proportion of the residue remained on the surface of the leaves (76–84% TRR in surface wash). The residues in or on the leaves could be extracted by acetonitrile/water (> 94% TRR). The principal component of the residue was the parent compound (59–78% TRR), followed by metabolite NK-1375 (13–22% TRR) and YT-1284 (0.3–0.6% TRR). A number of other metabolites were detected (0.2–2.2% TRR), but none reaching > 0.01 mg/kg eq.

The metabolism of ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cyclaniliprole in potato was studied following three foliar applications at 40 g ai/ha/application at a 14 days interval. Total radioactive residues (TRR) in potato foliage at DALA = 8 and 15 were 2.4 and 1.8 mg/kg eq for the phenyl label and 3.0 and 1.6 mg/kg eq for the pyrazole label, respectively. A high proportion of the residue remained on the surface of the leaves (44–57% TRR in surface wash). The residues in or on the leaves could be extracted by acetonitrile/water (> 90% TRR). The principal component of the residue was the parent compound (60–67% TRR), followed by metabolite NK-1375 (13–15% TRR). A number of other metabolites were detected, but none higher than 3.9% TRR. Residues in potato tubers were not investigated, because concentrations were below the trigger value of 0.01 mg/kg eq.

In summary, the metabolism in crops after foliar application is similar. In fruits, leafy vegetables and root and tuber vegetables the parent compound represents the principal part of the residue, followed by metabolite NK-1375. Metabolism follows two pathways. The major pathway is cyclisation by reaction between an oxygen moiety and a chloride moiety, giving the compound NK-1375 (pathway 1). The minor pathway is N-de-alkylation of cyclaniliprole and loss of the 1-cyclopropylethyl group on the nitrogen atom in the amide moiety in the side chain yielding the corresponding primary amide YT-1284 (pathway 2).

In general, metabolism between plants and rat is not similar, though some NK-1375 was found in fat, it was not observed in any other animal tissues. The plant metabolite YT-1284 (minor metabolite 0.3–3.9% TRR) was found in animal commodities (6.0–19% TRR).

Fate in rotational crops

Metabolism of cyclaniliprole was investigated in one confined rotational crop study and two field rotational crops studies.

In the confined rotational crop study ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cyclaniliprole was applied to bare sandy loam soil at a rate of 98–110 g ai/ha under indoor conditions. Rotational crops (wheat, lettuce and carrot) were sown at 30, 120 and 365-day plant back intervals (PBI). Total radioactivity in rotational crops were 0.001 mg/kg eq in lettuce, carrot root, carrot foliage and wheat grain at all pant back intervals, except for carrot foliage (0.002 mg/kg eq) at PBI 30 days. Total radioactivity in wheat forage was 0.018 mg/kg eq (PBI 30 days), 0.028 mg/kg eq (PBI 120 days) and

0.015 mg/kg eq (PBI 365 days). In wheat hay, total radioactivity decreased from 0.030 mg /kg eq (30-day PBI) to 0.017 mg/kg eq (365-day PBI). Total radioactivity in wheat straw decreased from 0.058 mg/kg eq (30-day PBI) to 0.029 mg/kg eq (365-day PBI).

In wheat forage, hay and straw parent was the principal component and ranged from 67% to 90% TRR at the three plant back intervals. Metabolite NK-1375 was found at a maximum of 14% TRR in forage (30-day PBI). NK-1375 in forage, hay and straw at other plant back intervals ranged from not detectable to 3.5% TRR.

From these data, the Meeting concluded that cyclaniliprole can be taken up from the soil under confined conditions even after long plant back intervals (365 days) in cereals, but it does not lead to detectable residues in leafy vegetables or root and tuber vegetables. No additional metabolites were observed in confined rotational crops indicating the same metabolic pathway as primary crops.

The Meeting received two field rotational crop studies to investigate the actual uptake of residue from soil.

In the first field rotational crop study at three different locations in the EU cyclaniliprole was applied to tomatoes and peppers as primary crops with two applications of 40 g ai/ha, with an interval of 10–11 days. Wheat was planted as a rotational crop 29–32, 124–154 days after application. Cyclaniliprole was found at a maximum concentration equal to the LOQ of 0.01 mg/kg in four samples; twice in straw of wheat drilled at approximately 30 days after last application, once in straw of wheat drilled 124 days after last application and one in forage of wheat drilled 128 days after last application. In all other samples cyclaniliprole was below the LOQ of 0.01 mg/kg. NK-1375 was not detected in any of the samples.

In another field rotational crop study at six different locations in the USA cyclaniliprole was applied as foliar application to lettuce, soybean or wheat at 1× 300 g ai/ha. The last application was at BBCH 71–89 of the primary crops. The primary crops were mowed and plots were tilled or plots were disked before planting the rotational crop. The rotational crop wheat was sown 29–30, 119–127/147 or 263–366 days after application.

No residues of parent (< 0.01 mg/kg) were found in wheat grain. Parent was found in wheat forage and wheat straw at levels < 0.01–0.073 mg/kg (PBI 20–30 days), < 0.01–0.189 mg/kg (highest individual analytical value; PBI 119–127 days), and < 0.01–0.083 mg/kg (PBI 147–366 days). Metabolite NK-1375 was not found at levels > 0.01 mg/kg in any of the harvested commodities at any of the rotations, except in wheat straw at the 127 day plant back interval on one location in the USA.

The dose rate of 1×300 g ai/ha covers the current maximum seasonal rates.

The Meeting concluded that residues due to crop rotation are not expected in leafy vegetables, root and tuber vegetables, cereal grain and leaves of root and tuber vegetables. No data are available for oilseeds and pulses. Residue levels due to crop rotation in wheat forage and straw can be expected.

Animal metabolism

The Meeting received results of metabolism studies in laboratory animals (rats), lactating goats and laying hens.

Metabolism in laboratory animals was summarised and evaluated by the WHO panel of the JMPR in 2017.

One lactating goat per radiolabel was dosed orally once daily for five consecutive days with a capsule containing ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cylcaniliprole. The actual dose levels were 12.3 ppm and 11.2 ppm for the phenyl and pyrazole labelled cyclaniliprole, respectively. The total overall recoveries of radioactivity were 81–87% of the cumulative dose following sacrifice at 23 hours after the last dose for both labels. The majority of the radioactivity was recovered in the faeces (68/59% TAR, phenyl/pyrazole). The remainder of the dose was recovered in urine (5.1/6.6% TAR, phenyl/pyrazole), GI tract contents (5.4%/9.5% TAR), and cage wash (0.2%/0.3% TAR). Total recovered radioactivity in milk and tissues was 8.8%/6.0% TAR, respectively.

The highest radioactivity concentrations in edible tissues were found in the liver (1.5/1.3 mg/kg eq), fat (0.86/0.79 mg/kg eq), and kidneys (0.58/0.55 mg/kg eq) followed by muscle (0.12/0.12 mg/kg eq). Total radioactive residues in milk from the phenyl label dosed goat reached a plateau concentration of approximately 0.12–0.14 mg/kg eq by 96–119 hours after dosing. Total radioactive residues in milk from the pyrazole label dosed goat reached a plateau concentration of approximately 0.081–0.091 mg/kg eq following 72 hours after dosing.

Following solvent extraction with acetonitrile, residue extractabilities in tissues and milk ranged from 84% (kidney) to 100% (fat).

Parent was identified in milk and all goat tissues at levels of 71/58% TRR in milk (0.094/0.048 mg/kg eq), 33/30% TRR (0.48/0.40 mg/kg eq) in liver, 30/19% TRR (0.17/0.10 mg/kg eq) in kidney, 44/23% TRR (0.052/0.027 mg/kg eq) in muscle and 76/44% TRR (0.67/0.31 mg/kg eq) in fat. The most significant metabolites identified in all tissues and milk were NSY-28, ranging from 5.2% TRR (milk) to 53% TRR (kidney) and metabolite YT-1284, ranging from 6.0% TRR (fat) to 21% TRR (milk). Levels of NSY-28 in kidney (0.21–0.29 mg/kg eq), muscle (0.055 mg/kg eq) and liver (0.42 mg/kg eq) were higher than those of the parent compound. Other metabolites (including metabolite NSY-27) were found at levels below 10% TRR.

Five laying hens per radiolabel were dosed orally once daily for 14 consecutive days with a gelatine capsule containing ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cyclaniliprole at mean daily doses in the dry feed of 11.03 and 10.8 ppm for the phenyl or pyrazole label, respectively. Hens were euthanized 12 hours after the last dose. Total recovered radioactivity amounted to 96% and 98% of the administered dose for the phenyl and pyrazole radiolabelled forms, respectively. The majority of the radioactivity was recovered in excreta (92%/93%, phenyl/pyrazole), while only low levels were found in eggs (2.0/2.5% TAR, phenyl/pyrazole) and tissues (0.8/0.7% TAR, phenyl/pyrazole).

The highest radioactivity concentrations were found in liver (1.7/1.5 mg/kg eq, phenyl/pyrazole), followed by fat (0.34/0.27 mg/kg eq, phenyl/pyrazole), skin (0.27/0.30 mg/kg eq, phenyl/pyrazole) and muscle (0.072/0.067 mg/kg eq, phenyl/pyrazole). Total radioactive residues in whole egg achieved a plateau concentration of 0.62–0.67 mg/kg eq after 8–9 days of dosing. Total radioactive residues in egg whites and egg yolk separately were not determined.

Following solvent extraction with acetonitrile, residue extractabilities were > 76% TRR for eggs and tissues.

Parent was identified at levels of 4.4–12% TRR (0.073–0.17 mg/kg eq) in liver, 9.7–16% TRR (0.006–0.011 mg/kg eq) in muscle, 21–23% TRR (0.15–0.16 mg/kg eq) in eggs, 44–58% TRR (0.15–0.16 mg/kg eq) in fat and 26–30% TRR (0.069–0.090 mg/kg eq) in skin. The most significant metabolite in all tissues and eggs was NSY-28 (54–63% TRR 0.38–0.42 mg/kg eq) in eggs, 63–56% TRR (0.82–1.0 mg/kg eq) in liver; 27–26% TRR (0.069–0.093 mg/kg eq) in fat; 38–47% TRR (0.10–0.14 mg/kg eq) in skin; 27–48% TRR (0.019–0.033 mg/kg eq) in muscle, followed by YT-1284 (4.0% TRR in eggs to 28% TRR in muscle). Levels of NSY-28 were higher than those of the parent compound in eggs, skin, muscle and liver.

In summary, metabolism observed in lactating goats and laying hens arose via N-de-alkylation of cyclaniliprole and loss of the 1-cyclopropylethyl group on the nitrogen atom in the amide moiety in the side chain yielding the corresponding primary amide, YT-1284. This metabolite is subjected to cyclization by reaction between two amide moieties in YT-1284 giving the quinazoline compound, NSY-28.

The major compounds identified in goat, hen tissues, milk or eggs are: parent and NSY-28, followed by YT-1284. Parent and NSY-28 comprise a significant part of the residue in tissues, milk and eggs. Significant additional contributions (> 10% TRR) are found for metabolite YT-1284 in all tissues and milk.

In general, metabolism between goat, hen and rat is similar.

Environmental fate in soil

The Meeting received information on aerobic metabolism in soil.

Aerobic metabolism and degradation in soil of ^{14}C-phenyl-cyclaniliprole or ^{14}C-pyrazole-cyclaniliprole was investigated in two studies at an application rate of 0.2 mg/kg, equivalent to a field rate of 150 g ai/ha in both studies. Cyclaniliprole was the major component at all times in both studies. The calculated DT_{50}s were > 692 days at 20 °C and 482–638 days at 35 °C.

The Meeting concluded that cyclaniliprole is very persistent.

Environmental fate in water-sediment systems

Not applicable.

Methods of analysis

The Meeting received description and validation data for analytical methods for the determination of cyclaniliprole related residues in plant and animal commodities.

The existing multi-residue method QuEChERS was submitted as an enforcement/monitoring method for the determination of parent compound and metabolite NK-1375 in plant commodities. The Meeting considers validation sufficient for plant commodities with high acid content, high water content, high starch content, high oil content. The LOQ was 0.01 mg/kg each for parent compound and metabolite NK-1375 in each matrix.

Several other LC-MS/MS methods were submitted for the determination of parent and its metabolite NK-1375 and degradation products BPQO, BCPBA and YT-1327 in plant material. Crop commodities were extracted with acetonitrile and cleaned up with SPE. The extraction efficiency of the residue analytical method was compared to the extraction efficiency of the analytical method used in the metabolism study. The extraction efficiency was 84% for total residues (cyclaniliprole and NK-1375) in surface washed lettuce. All analytical methods were considered fit for purpose with LOQs of 0.01 mg/kg for individual analytes. This procedure was not followed for the degradation products, but the submitted method was validated in several processed commodities derived from tomatoes and grapes.

A similar LC-MS/MS method was submitted for the determination of parent and its metabolites NK-1375, NSY-27, NSY-28 and YT-1284 in milk, eggs and animal tissues. Animal commodities were extracted twice with acetonitrile and subsequently partitioned against hexane. SPE clean-up is performed prior to LC-MS/MS analysis. The extraction efficiency of the residue analytical method was compared to the method used in the metabolism studies. The extraction efficiency for acetonitrile ranged from 70% to 93% in liver, milk and eggs as shown by a radio-validation study in goat milk and liver and hen egg and liver. The analytical method was considered fit for purpose with LOQs of 0.01 mg/kg for the individual analytes.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the storage stability of parent and metabolite NK-1375 in raw and processed plant commodities and of parent, NK-1375, NSY-27, NSY-28 and YT-1284 in animal commodities.

Storage stability studies showed that cyclaniliprole and metabolite NK-1375 were stable for at least 18 months at -20 °C in crop commodities representative of the high water, high acid, high starch, high protein and high oil commodity groups.

Cyclaniliprole and metabolite NK-1375 were stable for at least 18 months at -20 ºC in wine. No storage stability data were provided for the degradation products BPQO, BCPBA and YT-1327 found after some processing procedures.

Cyclaniliprole and the metabolites NK-1375, NSY-27, NSY-28 and YT-1284 were found to be stable at -20 ºC for at least 39 days in bovine liver, kidney, muscle, and fat. Stability data in metabolism studies show that no further degradation occurs for another 2–3 months.

Definition of the residue

In primary crops (apple, lettuce and potato), parent compound represented the principal part of the residue in most crop commodities ranging from 40% TRR to 78% TRR (0.015–9.7 mg/kg eq). The major metabolite in these crops was NK-1375 with concentrations ranging from 13–29% TRR (0.009–0.113 mg/kg eq). Metabolite YT-1284 was observed in apples (1.0–3.9% TRR (0.001–0.006 mg/kg eq)) and lettuce (0.3–0.6% TRR (0.002–0.003 mg/kg eq)).

In confined rotational crops no residues (< 0.001 mg/kg eq) were found in lettuce, carrot and wheat grain. Parent was the principal component in wheat forage, hay and straw (67–90% TRR, 0.012–0.044 mg/kg eq). Metabolite NK-1375 was detectable at a lower but significant level only at 30 day PBI in forage (14% TRR, 0.003 mg/kg eq) and was less than 10% TRR in wheat hay and straw and other PBIs (<3.5% TRR, < 0.001 mg/kg eq). In field rotational crops with wheat in rotation on nine locations in the USA and EU, NK-1375 was found once at detectable levels in wheat straw (0.013 mg/kg).

Cyclaniliprole is found in every primary crop commodity and is considered suitable as marker compound. The Meeting noted that suitable analytical methods exist to measure cyclaniliprole and its metabolites in plant commodities. The Meeting decided to define the residue for enforcement/monitoring as parent only.

Apart from cyclaniliprole, NK-1375 was found at significant levels in primary crops (pome fruit, stone fruit, grapes, brassica's, fruiting vegetables, leafy vegetables, tea) if the application rate is high enough. In the majority of these crops the metabolite accounted for 10–30% of the total residue. The JMPR 2017 received toxicological data for the metabolite NK-1375, showing that this compound is no more toxic than parent and that it was detected in rat metabolism studies.

Processing studies showed that hydrolysis under very specific conditions can lead to the formation of degradation products YT-1327, BCPBA and BPQO and might be relevant for the residue definition for processed commodities formed after sterilisation. Processing studies in grapes, apples, peaches and tomatoes show that even at exaggerated application rates, residues of these degradation products were below the LOQ, except for tomato paste where residues of YT-1327, BCPBA and BPQO were found, each accounting for less than 3% of parent. Considering that these levels were found at 6.5 times the highest GAP, the resulting residues at the critical GAP would be expected to be below the LOQ in all kinds of processed commodities and need not to be considered for the residue definition for dietary risk assessment.

In addition to the parent compound, NK-1375 is the only compound which might be relevant for the plant residue definition for dietary risk assessment. The Meeting decided to define the residue for dietary risk assessment for plant commodities as parent and NK-1375.

In animal metabolism studies the major compounds identified in goat, hen tissues, milk or eggs are: parent, NSY-28 and YT-1284. Parent was identified at levels ranging from 9.7% TRR to 76% TRR (0.006–0.673 mg/kg eq) in the different animal commodities. Metabolite NSY-28 was identified at levels of 5.2–63% TRR (0.007–1.049 mg/kg eq) in hen and goat commodities. Metabolite YT-1284 was identified at levels of 4.0–28% TRR (0.011–0.25 mg/kg eq) in hen and goat tissues, eggs and milk. In dietary feeding studies with the maximum dietary feeding level (11.6 ppm) slightly below the median dietary burden (12 ppm feed), metabolite YT-1284 was either not detectable or below the LOQ at the highest dose level. NSY-28 was detected in liver (0.014–0.032 mg/kg), kidney and fat (< 0.01–0.014 mg/kg) at the 11.6 ppm feeding level, representing 23–56% of the total residue in liver and max 12% of the total residue in kidney and fat.

Cyclaniliprole parent is found in every animal commodity and is therefore a suitable marker compound. The Meeting decided to define the residue for enforcement as parent only.

The log K_{ow} for cyclaniliprole is 2.0–2.8. The goat and hen metabolism studies showed a tendency of the parent compound to partition into the fat tissues; levels of cyclaniliprole found in goat fat were at least 6 times higher than residues in goat muscle. A similar, even more pronounced pattern (ratio 13.5:1) is observed for hen fat and muscle. The ratio of parent found in the fat and aqueous fraction of milk was at least 53:1. The dairy feeding study also showed a tendency to partition into fat. The Meeting considers the residue to be fat soluble.

Apart from cyclaniliprole, metabolites NSY-28 and YT-1284 were found at significant levels in livestock commodities and were considered for their relevance for the residue definitions. Levels of YT-1284 ranged from 4 to 28% in the various animal tissues in the two metabolism studies, but was not detected in any tissues in the dairy feeding study and therefore not expected to contribute to the dietary intake. Though significant levels of NSY-28 are found in liver and fat the metabolite is not expected to contribute significantly to the overall dietary intake via consumption of edible offal and fat in chronic intake assessment. Furthermore, the toxicity of these metabolites is considered to be covered by toxicity studies on cyclaniliprole since each of the metabolites was detected in the rat. The Meeting noted that suitable analytical methods exist to measure cyclaniliprole and its metabolites in animal commodities.

The Meeting decided to define the residue for dietary risk assessment for animal commodities as parent only.

The Meeting recommended the following residue definitions for cyclaniliprole:

Definition of the residue for compliance with the MRL for plant and animal commodities: *cyclaniliprole.*

Definition of the residue for dietary risk assessment for plant commodities: *cyclaniliprole + 3-bromo-2-((2-bromo-4H- pyrazolo[1,5-d]pyrido[3,2-b]-[1,4]oxazin-4-ylidene)amino)-5-chloro-N-(1-cyclopropylethyl)benzamide (NK-1375), expressed as cyclaniliprole equivalents.*

The molecular weight conversion factor to express NK-1375 in cyclaniliprole equivalents = 1.064.

Definition of the residue for dietary risk assessment for animal commodities: *cyclaniliprole*

The Meeting considers the residue to be fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trials data for cyclaniliprole on apple, pear, cherry, plum, peach, apricot, nectarine, grapes, head cabbages, Brussels sprouts, broccoli, cauliflower, cucumber, summer squash, melon, tomato, pepper, lettuces, spinach, mustard greens, kale, soybeans, almond, pecan, and tea.

Residues for maximum residue estimation are expressed in mg cyclaniliprole/kg. Residues for dietary risk assessment include parent cyclaniliprole and metabolite NK1375. The totals (sum of the mean of parent and NK-1375) are expressed as parent equivalents by applying a conversion factor of 1.064 to NK-1375. Levels of NK-1375 are generally not detectable if parent concentrations are < 0.01 mg/kg. Field trials resulting in significant residue levels show a maximum contribution of NK-1375 of approximately 30% to the total residue. Therefore, if parent is at or below LOQ the metabolite is < 0.01 mg/kg and the total is calculated as < 0.01 mg/kg.

For several crops (pome fruit (USA), stone fruit (USA), brassica's (USA), cucurbits (USA) and peppers and tomatoes (EU) field trials were performed with and without the use of an adjuvant. These trials demonstrate that the adjuvant does not influence the residue level.

None of the submitted trials from Canada and the USA matched the critical GAPs from the USA with respect to number of applications, retreatment intervals, and/or application rates. However, for some trials, the total seasonal rates were within ±25% of the maximum seasonal rates allowed on the label and the PHIs matched. To explore whether these trials could be used to evaluate maximum residue levels, the Meeting developed a simple tool to compare anticipated residues from at-GAP use

patterns with those from the use patterns used in the trials. The tool uses application rates, retreatment intervals, PHI, and half-life estimates to model residues in crops at harvest. If modeled residues were within ±25% for these scenarios, then the Meeting decided that the trials could be used to estimate maximum residue levels.

In implementing the tool, the Meeting used the submitted residue decline trials to derive crop(group) specific median half-life estimates; at least three decline trials, each with at least 4 time points, with the first time point well above LOQ and the following two time points at least at or above the LOQ. If the decline data were not sufficient to derive a reasonably robust median half-life estimate, or total application rates were not within ±25% of the maximum seasonal rate allowed on the label, then the tool was not used to evaluate the suitability of the trials.

Pome fruit

Field trials involving apples and pears were performed in the USA and Canada, EU (the Netherlands, Germany, United Kingdom, France, Italy and Spain) and Australia. Field trials involving apples performed in the EU (2 × *ca*. 40 g ai/ha, RTI 13–15 days, PHI 14 days) and field trials involving apples performed in Australia (2×*ca*. 30–300 g ai/ha, interval 13–15 days, PHI 28 days) did not match any GAP.

The critical GAP for pome fruit is the GAP from the USA: 1×60 + 3×80 g ai/ha, to reach the seasonal maximum rate of 300 g ai/ha, 10-day RTI, 7-day PHI.

Field trials with apples or pears from USA and Canada (3×59–107 g ai/ha, RTI 13–15 days, PHI 7 days) differed from the critical GAP with regard to the number of applications, the application rate and the retreatment interval. Comparison of application scenarios of the trials with the critical GAP, using the tool described earlier and applying a calculated median $t_{1/2}$ of 12 days (n=16) for pome fruit, show that the expected residues are similar (2.3% deviation). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in apples in ranked order were (n=16): 0.013, 0.023, 0.027, 0.035, 0.037, 0.046, 0.049, 0.054, 0.054, 0.055, 0.058, 0.068, 0.068, 0.10, 0.10, and 0.13 mg/kg cyclaniliprole.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in apples were (n=16): 0.023, 0.033, 0.038, 0.046, 0.053, 0.056, 0.059, 0.065, 0.065, 0.067, 0.073, 0.079, 0.084, 0.12, 0.13, and 0.17 mg/kg parent equivalents.

For estimation of the maximum residue level the ranked order of cyclaniliprole residues in pears were (n=9): 0.037, 0.060, 0.069, 0.095, 0.097, 0.11, 0.13, 0.14, and 0.14 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in pears were (n=9): 0.051, 0.070, 0.081, 0.11, 0.12, 0.12, 0.14, 0.16, and 0.16 mg/kg parent equivalents.

The datasets of apples and pears are of the same population (Mann-Whitney test). The Meeting decided to combine the data to estimate a maximum residue level for pome fruit. The combined cyclaniliprole residues in apples and pears were (n=25): 0.013, 0.023, 0.027, 0.035, 0.037, 0.037, 0.046, 0.049, 0.054, 0.054, 0.055, 0.058, 0.060, 0.068, 0.068, 0.069, 0.095, 0.097, 0.10, 0.10, 0.11, 0.13, 0.13, 0.14, and 0.14 mg/kg.

For dietary risk assessment cyclaniliprole residues include metabolite NK-1375 and the combined data in apples and pears were (n=25): 0.023, 0.033, 0.038, 0.046, 0.051, 0.053, 0.056, 0.059, 0.065, 0.065, 0.067, 0.070, 0.073, 0.079, 0.081, 0.084, 0.11, 0.12, 0.12, 0.12, 0.13, 0.14, 0.16, 0.16, 0.17 mg/kg equivalents.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.073 mg/kg parent equivalents for pome fruit.

The Meeting estimated a median residue of 0.060 mg/kg (parent only) for animal dietary burden calculations.

Stone fruit

Cherries

Field trials involving cherries were performed in the USA, Canada and Japan. Field trials in Japan (2×ca. 100 g ai/ha, interval 7 days, PHI 1–21 days) did not match any GAP.

The critical GAP for cherries is the GAP for stone fruit from the USA: 1×60 + 3×80 g ai/ha, to meet the maximum rate of 300 g ai/ha/season, 7-day RTI, 7-day PHI.

Field trials with cherries from the USA and Canada (3×100 g ai/ha, interval 6–8 days, PHI 7 days) differed from the critical GAP with regard to the number of applications and the application rate. Comparison of application scenarios of the trials with the critical GAP, using the tool described earlier and applying a calculated median $t_{1/2}$ of 7.2 days (n=15 from various stone fruit crops) for stone fruit, show that the expected residues are similar (max +18.4% deviation). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in cherries in ranked order were (n=15): 0.010, 0.016, 0.082, 0.097, 0.13, 0.13, 0.14, 0.14, 0.18, 0.24, 0.28, 0.30, 0.33, 0.44 and 0.56 mg/kg.

Residue levels in the field trials from the USA and Canada were determined in flesh without stone. The Japanese trials on cherries show that the contribution of the pit to the weight of the whole fruit is approximately 10%. Correction of the residue levels using this weight/weight ratio would lead to the same maximum residue level.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 as measured in flesh were (n=15): 0.021, 0.027, 0.10, 0.11, 0.14, 0.14, 0.16, 0.17, 0.19, 0.26, 0.32, 0.34, 0.34, 0.48 and 0.61 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.9 mg/kg and an STMR of 0.17 mg/kg parent equivalents for the subgroup cherries.

Plums

Field trials involving plums were performed in the USA, Canada and EU (Netherland, Germany, United Kingdom, France, Italy and Spain). Field trials involving plums performed in the EU (2×ca. 40 g ai/ha, RTI 13–15 days, PHI 13–15 days) did not match any GAP.

The critical GAP for plums is the GAP for stone fruit from the USA: 1×60 + 3×80 g ai/ha, to reach a maximum rate of 300 g ai/ha/season, 7-day RTI, 7-day PHI.

Field trials with plums from USA and Canada (3×40–102 g ai/ha, interval 6–8 days, PHI 7 days) differed from the critical GAP with regard to the number of applications and the application rate. Comparison of application scenarios of the trials with the critical GAP using the tool described earlier and applying a calculated median $t_{1/2}$ of 7.2 days (n=15 from various stone fruit crops) for stone fruit show that the expected residues are similar (max +18.4% deviation). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in plums in ranked order were (n=7): 0.019, 0.019, 0.024, 0.056, 0.062, 0.065, and 0.091 mg/kg.

Residue levels were determined in flesh without stone. The residue data from field trials in Europe show that the ratio of the residue levels in flesh versus whole fruit range between 0.86 and 0.97. Though these ratios are based on very low residue levels, still an overestimation of approximately 10% is anticipated. Correction with this factor would lead to the same maximum residue level.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in plums were (n=7): 0.030, 0.03, 0.035, 0.067, 0.075, 0.076, and 0.11 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR value of 0.067 mg/kg parent equivalents for the subgroup plums.

Peaches (including apricots and nectarines)

Field trials involving peaches were performed in the USA and Canada. Field trials involving apricots and peaches were performed in the EU (Spain, Southern France, Italy, Germany, Hungary and Poland). Field trials involving apricots and peaches performed in the EU (2×ca. 40 g ai/ha, RTI 13–15 days, PHI 13–15 days) did not match any GAP.

The critical GAP for apricots, peaches, nectarines is the GAP for stone fruit from the USA: 1×60 + 3×80 g ai/ha, max rate of 300 g ai/ha/season, 7-day RTI, 7-day PHI.

Field trials with peaches from the USA and Canada (3×55–103 g ai/ha, RTI 6–8 days, PHI 7 days) differed from the critical GAP with regard to the number of applications and the application rate. Comparison of application scenarios of the trials with the critical GAP using the tool described earlier and applying a calculated median $t_{1/2}$ of 7.2 days (n=15 from various stone fruit crops) for stone fruit show that the expected residues are similar (max +18.4% deviation). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in peaches in ranked order were (n=12): 0.023, 0.041, 0.045, 0.050, 0.051, 0.054, 0.064, 0.081, 0.094, 0.11, 0.16, and 0.19 mg/kg.

Residue levels were reported on a flesh basis. The residue data from supervised field trials in Europe show that the weight ratio's flesh/whole fruit range between 0.85 and 0.96. Though these ratios are based on very low residue levels, still an overestimation of approximately 10% is anticipated. Correction with this factor would lead to the same maximum residue level.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in peaches were (n=12): 0.034, 0.056, 0.058, 0.061, 0.062, 0.065, 0.078, 0.092, 0.10, 0.12, 0.17, 0.20 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.0715 mg/kg parent equivalents for peach. The Meeting decided the maximum residue level and STMR can be extrapolated to the whole subgroup of peaches (including apricots and nectarines).

Grapes

Field trials involving grapes were performed in the USA, Canada, EU (Italy Spain, Greece, Germany, France), and Japan. The residue field trials involving table and wine grapes in the EU (2×ca. 35 g ai/ha, interval 13–15 days, PHI 3 days) did not match any GAP. Similarly residue field trials on grapes performed in Japan (2×ca. 80 g ai/ha, interval 7 days, PHI 1–21 days) did not match any GAP.

The critical GAP for grapes is the GAP for small fruit (vine climbing fruit) from the USA: 1×60 + 3×80 g ai/ha, to reach the maximum rate of 300 g ai/ha/season, 7-day RTI, 7-day PHI.

Field trials with grapes from the USA and Canada (3×97–105 g ai/ha, interval 6–8 days, PHI 7 days) differed from the critical GAP with regard to the number of applications and the application rate. Comparison of application scenarios of the trials with the critical GAP using the tool described earlier and applying a calculated median $t_{1/2}$ (n=15) of 11 days for grapes show that the expected residues are similar (max +13.9% deviation). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in grapes in ranked order were (n=15): 0.025, 0.044, 0.048, 0.076, 0.11, 0.12, 0.12, 0.14, 0.14, 0.16, 0.20, 0.24, 0.33, 0.39 and 0.51 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in grapes were (n=15): 0.036, 0.055, 0.059, 0.092, 0.13, 0.13, 0.15, 0.15, 0.17, 0.22, 0.25, 0.28, 0.44, 0.48, and 0.59 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.8 mg/kg and an STMR of 0.15 mg/kg parent equivalents for grapes.

The Meeting estimated a median residue of 0.14 mg/kg (parent only) for animal dietary intake calculations.

Leek

Though the Meeting received a GAP for leek, no supporting field residue trials were submitted.

Brassica vegetables (except Brassica leafy vegetables)

Flowerhead Brassicas

Field residue trials on broccoli were performed in the USA and Canada and in Europe (Germany, United Kingdom, France, Italy, and Spain). Residue trials on cauliflower were performed in Europe (Germany, the Netherlands, France, Italy, and Spain). The trials performed in the EU did not match any GAP.

The critical GAP flower head brassica's is the USA GAP for brassica's: 4×60 g ai/ha, max total rate of 240 g ai/ha/season, 5-day RTI, 1-day PHI.

Field trials with broccoli from the USA and Canada (3×61–87 g ai/ha, interval 6–8 days, PHI 1 day) differed from the critical GAP with regard to the number of applications and the application rate, but were comparable using the tool described earlier. Modelling with the calculated median $t_{1/2}$ of 1.8 days (n=5) for brassica's, show that the expected residues are similar (-8% to +22% deviation, comparing the 3×60 and 3×80 g ai/ha/application patterns from the trials to the 4×60 g ai/ha/application GAP pattern). The Meeting concluded that the supervised residues trials could be used for estimation of a maximum residue level. Cyclaniliprole residues ranked order were (n=10): 0.11, 0.12, 0.18, 0.20, 0.34, 0.37, 0.41, 0.42, 0.47, and 0.66 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 were (n=10): 0.12, 0.13, 0.19, 0.23, 0.38, 0.38, 0.42, 0.49, 0.54, and 0.71 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 1.0 mg/kg and an STMR of 0.38 mg/kg parent equivalents for flower head brassicas.

Head Brassicas

Field trials conducted on head cabbage were performed in the USA and Canada and in the EU (Germany, Northern and Southern France, Italy and Spain). The trials performed in the EU did not match any GAP.

The critical GAP for head cabbages is the USA GAP for brassicas: 4×60 g ai/ha, max total rate of 240 g ai/ha/season, 5-day RTI, 1-day PHI.

Field trials with head cabbages from the USA and Canada (3×61–102 g ai/ha, RTI 6–8 days, PHI 1 day) differed from the critical GAP with regard to the number of applications and the application rate, but were comparable using the tool described earlier. Modelling with the calculated median $t_{1/2}$ of 1.8 days (n=5) for brassicas, show that the expected residues are similar (max -8% to +22% deviation up to the 3×80 g ai/ha, comparing the 3x60, 3x80 and x3x100 g ai/ha/application patterns from the trials to the 4×60 g ai/ha/application GAP pattern). The Meeting concluded that the supervised residues trials up to 3×80 g ai/ha/application could be used for maximum residue estimation. Cyclaniliprole residues in head cabbages in ranked order were (n=8): < 0.01, < 0.01, 0.014, 0.027, 0.082, 0.15, 0.32, and 0.39 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in head cabbages were (n=8): < 0.01, < 0.01, 0.025, 0.038, 0.094, 0.17, 0.34, and 0.42 mg/kg parent equivalents.

The Meeting estimated a maximum residue level for head cabbages of 0.7 mg/kg and an STMR of 0.066 mg/kg parent equivalents.

Residue trials on Brussels sprouts were performed in Europe (Germany, the Netherlands, United Kingdom, France, Italy, and Greece). The trials performed in the EU did not match any GAP.

Fruiting vegetables, Cucurbits

Cucumber and summer squash

Residue trials were conducted on cucumbers and summer squash in the USA and Canada.

The critical GAP for fruiting vegetables, cucurbits is the GAP from the USA for "cucurbit vegetables"; 4×60 g ai/ha, max 240 g ai/ha/season, 5-day RTI, 1-day PHI.

Field trials from the USA and Canada with cucumbers (3×76–84 g ai/ha, RTI 6–8 days, 1-day PHI) and summer squash (3×77–83, interval 6–8 days, PHI 1 day) differed from the critical GAP with regard to the number of applications, and/or the application rate and the retreatment interval, but were comparable using the tool described earlier. Modelling with the calculated median $t_{1/2}$ of 2.4 days (n=3) for cucurbits, show that the expected residues are similar (max +18%). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in cucumbers in ranked order were (n=9): < 0.01, < 0.01, 0.011, 0.013, 0.014, 0.018, 0.019, 0.024, and 0.025 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in cucumber were (n=9): < 0.01, < 0.01, 0.022, 0.024, 0.025, 0.029, 0.030, 0.035, and 0.036 mg/kg parent equivalents.

For estimation of the maximum residue level the ranked order of cyclaniliprole residues in summer squash were (n=9): < 0.01, < 0.01, 0.014, 0.016, 0.026, 0.028, 0.028, 0.033, and 0.046 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in summer squash were (n=9): < 0.01, < 0.01, 0.025, 0.027, 0.037, 0.039, 0.040, 0.043 and 0.057 mg/kg parent equivalents.

Since the datasets of cucumber and summer squash are similar, the Meeting decided to combine the two datasets of cucumber and summer squash.

For estimation of the maximum residue level the ranked order of the combined cyclaniliprole residues in cucumber and summer squash were (n=18): < 0.01, < 0.01, < 0.01, < 0.01, 0.011, 0.013, 0.014, 0.014, 0.016, 0.018, 0.019, 0.024, 0.025, 0.026, 0.028, 0.029, 0.033, and 0.046 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in cucumber and summer squash were (n=18): < 0.01, < 0.01, < 0.01, < 0.01, 0.022, 0.024, 0.025, 0.025, 0.027, 0.029, 0.030, 0.035, 0.036, 0.037, 0.039, 0.040, 0.043, and 0.057 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.06 mg/kg and an STMR of 0.028 mg/kg parent equivalents for the subgroup of fruiting vegetables, cucurbits – cucumbers and summer squash.

Melons, pumpkins and winter squashes

Residue trials were conducted on melons in the USA and Canada.

The critical GAP for melons is the GAP from the USA for "cucurbit vegetables"; 4×60 g ai/ha, maximum 240 g ai/ha/season, 5-day RTI, 1-day PHI.

Field trials from the USA and Canada with melons (3×76–85, 6–8 day RTI, 1-day PHI) differed from the critical GAP with regard to the number of applications, and/or the application rate and the retreatment interval. Comparison of application scenarios of the trials with the critical GAP, using the tool described earlier and applying a calculated median $t_{1/2}$ (n=3) of 2.4 days for cucurbits,

show that the expected residues are similar (max +18% deviation). The Meeting concluded that the supervised residue trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in melons in ranked order were (n=10): 0.014, 0.017, 0.023, 0.039, 0.040, 0.042, 0.044, 0.051, 0.071, and 0.087 mg/kg.

In the absence on data on melons without peel residue levels used for estimation of the STMR and HR for melons are based on whole fruit. For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in melon were (n=10): 0.024, 0.028, 0.033, 0.050, 0.055, 0.055, 0.058, 0.063, 0.081, and 0.099 mg/kg parent equivalents for whole fruit.

The Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.055 mg/kg parent equivalents for the subgroup of fruiting vegetables, cucurbits – melons, pumpkins and winter squash.

Fruiting vegetables, other than Cucurbits

Indoor field trials involving application of cyclaniliprole on tomato and sweet pepper were performed in the EU (2×ca. 40 g ai/ha, interval 10–12 days, PHI 1–7 days). The indoor field trials did not match any GAP and were not further considered.

Tomatoes (outdoor)

Field trials involving outdoor applications of cyclaniliprole on tomatoes were performed in the EU, USA and Canada, and Japan. Trials performed in the EU (2×ca. 40 g ai/ha, 10–12 day RTI, 1–7 days PHI) and trials performed in Japan (2×111–141 g ai/ha, 7-day RTI, 1–21 day PHI) did not match any GAP.

The critical GAP for tomatoes is the USA GAP for "fruiting vegetables"; 4×60 g ai/ha, max 240 g ai/ha/season, 5-day RTI, 1-day PHI.

Field trials with tomatoes, including cherry tomatoes, from the USA and Canada (3×60–97 g ai/ha, 6–8 day RTI, 1-day PHI) differed from the critical GAP with regard to the number of applications, and/or the application rate and the retreatment interval. Comparison of application scenarios of the trials with the critical GAP, using the tool described earlier and applying a calculated median $t_{1/2}$ of 12 days (n=12) for tomatoes, show that the expected residues are similar (-23% to + 3% deviation of GAP, comparing both the 3×60 and 3×80 g ai/ha/application patterns from the trial to the 4×60 g ai/ha/application GAP pattern). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in tomato in ranked order were (n=22): 0.011, 0.013, 0.016, 0.018, 0.019, 0.024, 0.024, 0.025, 0.026, 0.027, 0.029, 0.030, 0.032, 0.032, 0.034, 0.037, 0.038, 0.040, 0.042, 0.043, 0.070, and 0.076 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 were (n=22): 0.022, 0.024, 0.029, 0.029, 0.030, 0.035, 0.035, 0.036, 0.036, 0.037, 0.040, 0.041, 0.042, 0.043, 0.045, 0.048, 0.049, 0.051, 0.053, 0.053, 0.080 and 0.10 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.041 mg/kg parent equivalents for cherry tomatoes and tomatoes.

The Meeting estimated a median residue of 0.0295 mg/kg (parent only) for animal dietary intake calculations.

The USA critical GAP for fruiting vegetables, other than cucurbits also covers egg plants. The Meeting decided the data could be used to extrapolate the maximum residue level and the STMR of tomato to the subgroup of eggplants.

Chili pepper, indoor

The critical GAP for chili pepper in the Republic of Korea is for 2 applications at 45 g ai/ha, 10 day RTI, 3-day PHI.

One indoor field trial with cyclaniliprole on chili peppers (2×45 g ai/ha, 7-day RTI, 3-day PHI) performed in the Republic of Korea matched this GAP within 25%. Cyclaniliprole residues in chili peppers are (n=1): 0.040 mg/kg.

The Meeting considered the data insufficient for estimating a maximum residue level for chili pepper based on these data.

Peppers (field)

Field trials involving outdoor applications of cyclaniliprole on sweet peppers were performed in the EU (n=16) and USA and Canada (n=12). The trials performed in the EU (2x ca 40 g ai/ha, 10–12 day RTI, 1–7 day PHI) did not match any GAP.

The critical GAP for sweet peppers is the USA GAP for "fruiting vegetables"; 4×60 g ai/ha, max 240 g ai/ha/season, 5-day RTI, 1-day PHI.

Field trials with bell (9 field trials) and non-bell (3 field trials) peppers from the USA and Canada (3×60–82 g ai/ha, 6–8 day RTI, 1-day PHI) differed from the critical GAP with regard to the number of applications, and/or the application rate and the retreatment interval. Comparison of application scenarios of the trials with the critical GAP, using the tool described earlier and applying a calculated median $t_{1/2}$ of 6.0 days (n=10) for peppers, show that the expected residues are similar (-20% to +7% deviation of GAP, comparing both the 3×60 and 3×80 g ai/ha/application patterns to the GAP pattern of 4×60 g ai/ha/application). The Meeting concluded that the supervised residues trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in peppers and non-bell peppers in ranked order were (n=12): 0.014, 0.019, 0.025, 0.041, 0.046, 0.048, 0.057, 0.068, 0.072, 0.077, 0.098, and 0.10 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 in sweet bell and non-bell peppers were (n=12): 0.025, 0.029, 0.035, 0.051[NB], 0.056, 0.059, 0.067[NB], 0.083, 0.094[NB], 0.096, 0.11, and 0.12 mg/kg parent equivalents.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.063 mg/kg parent equivalents for the subgroup peppers (excluding martynia, okra and roselle).

Chili peppers, dried

Based on the estimated maximum residue level of 0.2 mg/kg for the subgroup peppers (excluding Martynia, okra and Roselle) and applying a default processing factor of 10, the Meeting estimated a maximum residue level of 2 mg/kg for peppers, chili, dried, together with an STMR of 0.63 mg/kg parent equivalents (0.063 mg/kg×10).

Leafy vegetables

Leafy greens

Residue trials were conducted on head and leafy lettuce, spinach in the USA and Canada.

The critical GAP for lettuce, head, lettuce, leafy and spinach is the USA GAP for leafy vegetables (non-brassica's) of 4×60 g ai/ha, max 240 g ai/ha/season, 5 day-RTI, 1-day PHI. Field trials with lettuce, head (3×61–86 g ai/ha, 6–8 day RTI, 1-day PHI), lettuce, leafy (3×61–100 g ai/ha, 6–9 day RTI, 1-day PHI), and spinach (3×60–81 g ai/ha, 6–8 day RTI, 1-day PHI) from USA and Canada differed from the critical with regard to the number of applications, and/or the application rate and the retreatment interval. The available decline data were insufficient to estimate a median half-life and to use the tool described earlier to conclude whether the residue trials support the critical GAP (i.e. residues ± 25%).

The Meeting did not estimate a maximum residue level and STMR for leafy vegetables, subgroup leafy greens.

Brassica leafy vegetables

Field trials from Japan on Chinese cabbage (2×50–73, 6–8 day RTI, 1-day PHI) could not be matched to the Korean GAP of 2×45 g ai/ha, 10-day RTI, 14-day PHI. Field trials involving kale were conducted in the EU (2×25 g ai/ha, RTI 13–14 days, PHI 13/14 days). The trials could not be matched to any GAP.

The critical GAPs for Chinese cabbage and for kale fall within the USA GAP for brassicas (cole) leafy vegetables: 4×60 ga ai/ha, max 240 g ai/ha/season, 5-day RTI, 1-day PHI. No field trials on Chinese cabbage or kale according to this USA GAP were performed.

The critical GAP for mustard greens is the USA GAP for leafy vegetables (non-brassica's): 4×60 g ai/ha, max 240 g ai/ha/season, 5 day-RTI, 1-day PHI.

Field trials with mustard greens from the USA and Canada (3×60–81 g ai/ha, 6–8 day RTI, 1-day PHI) differed from the critical GAP with regard to the number of applications, and/or the application rate and the retreatment interval. Comparison of application scenarios of the trials with the critical GAP, using the tool described earlier and applying a calculated median $t_{1/2}$ of 2.5 days (n=7) for leafy vegetables, show that the expected residues are similar (-12% to +17% deviation of GAP, comparing both the 3×60 and 3×80 g ai/ha/application patterns to the 4×60 g ai/ha/application of the critical GAP). The Meeting concluded that the supervised residue trials could be used for estimation of the maximum residue level.

Cyclaniliprole residues in mustard greens were (n=5): 1.4, 3.0, 4.0, 4.1, and 5.9 mg/kg.

For the estimation of the dietary intake the ranked order of cyclaniliprole residues including metabolite NK-1375 were (n=5): 1.5, 3.5, 4.3, 4.4, and 6.2 mg/kg equivalents.

The Meeting estimated a maximum residue level of 15 mg/kg and an STMR of 4.3 mg/kg equivalents for mustard greens. The Meeting estimated a median and highest residue value of 4.0 and 6.5 mg cyclaniliprole/kg (highest individual value), respectively for mustard greens for livestock dietary burden calculations. The Meeting decided to extrapolate the maximum residue levels, STMR, median and highest residue value to the whole subgroup of brassica leafy vegetables.

Legume vegetables (soya bean, green)

Field trials (three trials) involving soya bean, immature (with pods) were conducted in Japan (2×38–50 g ai/ha, RTI 7 days, PHI 1–21 days). Without a supportive GAP the trials were not further considered.

Pulses (soya bean, dry)

Field trials involving soya bean, dried (six trials) were conducted in Japan (2×38–49 g ai/ha, RTI 6–8 days, PHI 1–21 days). Without a supportive GAP the trials were not further considered.

Tree nuts

Field trials involving almonds and pecans were performed in the USA.

The critical GAP for tree nuts is the USA GAP of 1×60 + 3×80 g ai/ha, to reach the max of 300 g ai/ha/season, 10 day-RTI, 30-day PHI.

Field trials with almonds and pecans from the USA and Canada (3×99–105 g ai/ha, 13–15 day RTI, 30-day PHI) differed from the critical GAP with regard to the number of applications, and/or the application rate and the retreatment interval. The available decline data were insufficient to estimate a median half-live and to use the tool described earlier to conclude whether the residue trials support the critical GAP (i.e. residues ± 25%).

The Meeting did not estimate a maximum residue level and STMR for almonds and pecans.

Tea

Field trials involving tea (six trials) were conducted in Japan (1×171–199 g ai/ha, PHI 3–21 days). Without a supportive GAP the trials were not further considered.

Animal feeds

Almond hulls

Field trials involving almond hulls were performed in the USA.

The critical GAP for tree nuts in the USA and Canada is for 3×80 and 1×60 g ai/ha, to reach the seasonal maximum rate of 300 g ai/ha at 10 day intervals and a PHI of 30 days.

Field trials with almonds and pecans from USA and Canada (3×99–105 g ai/ha, 13–15 day RTI, 30-day PHI) differed from the critical with regard to the number of applications, and/or the application rate and the retreatment interval. The available decline data were insufficient to estimate a median half-live and subsequently use the tool described earlier to conclude whether the residue trials support the critical GAP (i.e. residues ± 25%).

The Meeting did not estimate a median and highest residue for almond hulls.

Rotational crops

Based on results of the confined and field rotational crop studies the Meeting concluded that residues are not expected in leafy vegetables, root and tuber vegetables, cereal grain and leaves of root and tuber vegetables. No data are available for oilseeds and pulses.

The Meeting did not estimate maximum residue levels for rotational crops for human consumption.

Wheat (forage and straw)

Though the results of the confined and field rotational crops studies indicate that no residues occur in rotational crops for human consumption, the Meeting concluded that residues can be expected in wheat forage and wheat straw and decided to estimate median and highest residues for wheat forage and wheat straw.

Residue levels in wheat forage were highest at a plant back interval of 120 days. Furthermore, the USA label includes a plant back restriction of 30 days, indicating the 120 days represents the most realistic situation. Concentrations of cyclaniliprole in ranked order were (n=6): < 0.01 (4x), 0.019, and 0.026 mg/kg as received. For dietary burden calculation, NK-1375 does not need to be taken into account.

The Meeting estimated a median and highest residue level of 0.01 and 0.026 mg/kg on an as received basis for wheat forage.

Residue levels in wheat straw were highest at a plant back interval of 120 days (n=6): 0.011, 0.020, 0.024, 0.071, 0.12, 0.18 mg/kg cyclaniliprole as received.

The Meeting estimated a maximum residue level for wheat straw and fodder of 0.45 mg/kg (dw) based on a dry matter content of 88%. The Meeting estimated a median and highest residue of 0.0475 and 0.18 mg/kg (parent only) on an as received basis. Conversion to dry matter based on the dry matter content of 88% results in a median and highest residue level of 0.054 (dw) and 0.20 (dw) mg/kg (parent only), respectively for wheat straw and fodder.

These values for wheat forage and wheat straw and fodder were extrapolated to all other grain forages, straws and fodders in the group cereal grains.

Fate of residues during processing

High temperature hydrolysis

Degradation of [^{14}C] cyclaniliprole was studied under hydrolytical conditions at high temperatures in sterile aqueous buffers at pH4, 5 and 6 for periods of up to 60 minutes. Data showed that cyclaniliprole is not degraded during simulation of pasteurisation (pH 4, 90 °C, for 20 minutes). Data do show, however, that cyclaniliprole partly degrades into BPQO (11 %TAR) and YT-1327 (11% TAR) under baking, boiling and brewing conditions (pH 5, 100 °C, for 60 minutes). During sterilisation conditions (pH 6, 120 °C, for 20 minutes) BCPBA (23% TAR), BPQO (16% TAR) and YT-1237 (44% TAR) is formed. Though the hydrolysis study indicates that degradation products can be formed under specific processing conditions, these findings were not supported in the processing studies, except for tomato puree, where residues were found at concentrations up to 3% of the parent compound. The processing study was performed at an exaggerated dose level. The breakdown products are not expected to be detectable under normal use conditions.

Residues in processed commodities

Processing studies were undertaken for apples, peaches, tomatoes, plums, grapes, and tea. Two types of processing studies were performed; processing studies with spiked samples and with incurred residues. The spiked samples were not considered relevant for derivation of processing factors. The estimated processing factors derived from processing studies with incurred residues in combination with the estimated maximum residue levels and STMRs from supervised trials proposed STMR-Ps and median-P residues are summarised in the table below. MRLs in processed commodities are only proposed where they are higher than the MRL in the raw commodity. For estimation of the STMR-P the processing factors are based on parent + metabolite NK-1375. For MRL derivation and dietary burden calculation the processing factors are based on parent only (separate table).

Commodity	PF residue: parent + NK-1375	PF (median or best estimate)	STMR in RAC	STMR-P
Apples			0.073	
- juice, past.	0.13, < 0.33, < 0.5	< 0.33 (median, n=3)		0.024
Plums			0.067	
- dried prunes	3.7 [a]	3.7 (n=1)		0.25
Grapes			0.15	
- must	0.63 , 0.63, 0.71, 0.86	0.67 (median n=4)		0.10
- juice after pasteurisation	0.20, 0.12, 0.33, 0.38, 0.50, 0.71	0.36 (median, n=6)		0.54
- stored wine	0.14, 0.20 , < 0.33, 0.38, 0.040, 0.50	0.355 (median, n=6)		0.053
- raisins	< 0.14, 0.14, < 0.020, 0.50, 0.75, 0.75	[a]		[a]
Tomatoes			0.041	
- canned	< 0.14, < 0.17, < 0.2, < 0.5, < 0.5	0.2 (median, n=5)		0.008
- paste [b]	0.49, 0.50, 0.67, 1.57, 1.8, 2.5	1.12 (median, n=6)		0.046
- juice, pasteurised	< 0.5, 0.17, 0.8, 1.14, 1.5	0.8 (median, n=6)		0.033
- dried tomatoes	3, 3.2, 3.3, 5, 6	3.3 (median, n=5)		0.14

PF based on total cyclaniliprole; STMR-P is used for the long-term and short-term dietary exposure estimates and are based on the residue definition for dietary risk assessment.

[a] PF for raisins was not considered suitable without a plausible explanation why dilution instead of concentration occurred

[b] Values include PFs derived in European studies defining the processed product as puree. Since evaporation was used to concentrate the volume about 3 times, it was scaled under paste.

Commodity	PF residue: parent only	PF (median/best estimate)	Median residue in RAC	Median
Apple, wet pomace	3. 2	3.2 (n=1)	0.060	0.19
Grape, wet pomace	1.0, 1.17, 1.20, 1.64, 1.71, 3.0, 4.67, 8.0	1.7 (median n=8)	0.14	0.24
Grape, dry pomace	0.83, 3.07, 3.07, 2.83	3.1 (median, n=4)	0.14	0.43
Plums, dried	3.7	3.7 (n=1)	-	-
Tomato, dried	3.33, 3.75, 3.8, 4, 5.5	3.8 (median, n=5)	-	-
Tomato, wet pomace	0.67	0.67 (n=1)	0.0295	0.020
Tomato, dry pomace	22	22 (n=1)	0.0295	0.65

Median-P residues based on total cyclaniliprole, are used for dietary burden calculation

Total cyclaniliprole was shown to concentrate in prunes and dried tomatoes as well as in wet and dry pomace of apples and grapes. Despite the drying process, no concentration was observed in raisins. No clarification was provided. Since no breakdown of the compound is expected and drying of plums do lead to the expected increase in residue concentrations, the Meeting decided not to estimate an MRL or STMR for raisins.

The Meeting estimated a maximum residue level of 0.8 mg/kg (0.2 mg/kg×3.7=0.74 mg/kg) for prunes. The Meeting estimated a maximum residue level of 0.4 mg/kg (0.1 mg/kg×3.8 = 0.38 mg/kg) for dried tomatoes, using the processing factors based on parent only.

Livestock dietary burden

The Meeting estimated the dietary burden of cyclaniliprole in livestock on the basis of the diets (US/CAN, EU, Australia and Japan) listed in the OECD Feed Table 2013. Calculation from highest residue and median values (some bulk commodities) provide the levels in feed suitable for estimating maximum and highest residue levels while calculation from median values for feed is suitable for estimating STMR values for animal commodities

Some processed and forage commodities do not appear in the Recommendations Table (because no maximum residue level is needed), but they are used in estimating livestock dietary burdens. Those commodities are included in the list below. Almond hulls (AB0660) and soya beans were not included in the dietary burden calculation since they could not be matched to a GAP. In the rotational crops studies residues of cyclaniliprole were detected in wheat straw and forage. For the dietary burden calculation, these levels were widely extrapolated to the straw/hay (dry feed commodities) and forage (wet feed commodities) of the whole group of cereals grain crops. The input was based on the intake of parent only.

Codex classification	Commodity	Median residue (-P) (mg/kg) [a]	Highest residue (-P) (mg/kg) [a]
AB 0226	Apple pomace, wet (median 0.060 mg/kg ×PF 3.2)	0.19	-
AB 0269	Grape pomace, wet (median 0.14 mg/kg ×PF 1.7)	0.24	-
AV 0480	Kale forage (leaves) – based on the median and highest residue for brassica leafy vegetables (based on mustard greens dataset)	4.0	6.5
AB – no code	Tomato pomace, wet (median of 0.0295×PF 0.67 (n=1)) [a b]	0.020	-
AF – no code	Barley forage (30% DM)	0.01	0.026
AS 0640	Barley, hay (88% DM)	0.0475	0.18
AS 0641	Barley, straw (89% DM)	0.0475	0.18
AF/AS – no code	Corn, field, forage/silage (40% DM)	0.01	0.026
AS 0645	Corn, field, stover (83% DM)	0.0475	0.18
AF – no code	Corn, pop, stover (83% DM)	0.0475	0.18
AF – no code	Corn, sweet, forage (48% DM)	0.01	0.026
AF – no code	Maize (corn, sweet, stover) (83% DM)	0.0475	0.18
AF – no code	Millet, forage (30% DM)	0.01	0.026
AF – no code	Millet, hay (85% DM)	0.0475	0.18
AF 0646	Millet, straw (90% DM)	0.0475	0.18

AF 0647	Oat, forage (30% DM)	0.01	0.026
AS 0647	Oat, hay (90% DM)	0.0475	0.18
AF – no code	Oat, straw (90% DM)	0.0475	0.18
AS0469	Rice, straw (90% DM)	0.0475	0.18
AF0650	Rye, forage (30% DM)	0.01	0.026
AS0650	Rye, straw (88% DM)	0.0475	0.18
AF0651	Sorghum, grain, forage (35% DM)	0.01	0.026
AS – no code	Sorghum, grain, stover (88% DM)	0.0475	0.18
AF – no code	Triticale, forage (30% DM)	0.01	0.026
AF – no code	Triticale, hay (88% DM)	0.0475	0.18
AF – no code	Triticale, straw (90% DM)	0.0475	0.18
AF 0654	Wheat forage (25% DM)	0.01	0.026
AS 0654	Wheat, hay (88% DM)	0.0475	0.18
AS 0654	Wheat, straw (88% DM	0.0475	0.18

[a] levels for cereal straw, hay, and forage are presented on as received basis.

[b] Using the STMR-P of 0.76 mg/kg for tomato, dry pomace and assuming 80% for the dry matter content does not lead to a different outcome of the dietary burden calculation.

The dietary burden calculation of cyclaniliprole for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6. The calculations were made according to the livestock diets from US/CAN, EU, Australia and Japan in the OECD Feed Table 2013.

		Livestock dietary burden for cyclaniliprole (based on cyclaniliprole parent only) ppm of dry matter diet			
		USA/CAN	EU	Australia	Japan
Max	beef cattle	0.033	8.8	0.49	-
	dairy cattle	0.12	8.8	18	0.043
	poultry – broiler	-	-	-	-
	poultry – layer	-	0.015	-	-
Mean	beef cattle	0.0086	5.9	0.37	-
	dairy cattle	0.066	5.8	11	0.015
	poultry – broiler	-	-	-	-
	poultry – layer	-	0.05	-	-

Based on the calculations in the above table the Australian diets resulted in the highest maximum or mean beef or dairy cattle dietary burdens and would normally be used for maximum residue level estimates for mammalian meat and milk. The intake is driven by intake via kale (brassica leafy vegetables). No use on kale (or any other leafy vegetables) is included in the Australian registration application (under evaluation). Furthermore, the anticipated Australian orchard uses preclude rotational crops. For dietary burden calculation of the Australian diets only apple pomace was included. The European dietary burden was set at zero intake, because no uses in Europe are anticipated (active substance was withdrawn). As such no dietary burden for poultry is estimated for any diet, The remaining dietary burden is shown in the table below.

		Livestock dietary burden for cyclaniliprole (based on parent only), ppm of dry matter diet			
		USA/CAN	EU	Australia	Japan
Max	beef cattle	0.033	-	0.095	-
	dairy cattle	0.12 [a]	-	0.048	0.043

	poultry – broiler	-	-	-	-
	poultry – layer	-	-	-	-
Mean	beef cattle	0.009	-	0.095[c]	-
	dairy cattle	0.066 [b]	-	0.048	0.015
	poultry – broiler	-	-	-	-
	poultry – layer	-	-	-	-

[a] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian meat and milk.

[b] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian milk.

[c] Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian meat

Residues in animal commodities

The Meeting received a lactating dairy cow feeding study, which provided information on likely residue in animal tissues and milk from cyclaniliprole residues in animal diets.

Fifteen lactating Friesian cows were administered cyclaniliprole orally twice daily via capsules for 28–31 consecutive days at a feeding level of 0, 1.2, 3.5 and 11.6 ppm, corresponding with actual mean dose levels of 0, 4.9, 14 and 49 mg/animal/day (equivalent with 0, 0.0075, 0.021, and 0.075 mg/kg bw/day, respectively).

Cyclaniliprole-derived residues above the limit of quantitation were found only incidentally in whole milk samples. Residues were generally below the limit of quantitation (< 0.01 mg/kg) throughout the treatment and depuration periods. No residues of cyclaniliprole were detected above the limit of quantitation (0.01 mg/kg) in any samples taken on Days 21 and 28 in skimmed milk. Dose-related quantifiable residues were found in cream in samples taken on Days 21 and 28 and comprised of parent compound only. The mean values were 0.02 mg/kg and 0.078 mg/kg in the mid and highest dose group while the was 0.011 mg/kg at the lowest feeding level (1.2 ppm).

Concentrations of cyclaniliprole at the lowest feeding level of 1.2 ppm were either not detectable (muscle) or < 0.01 mg/kg, except for kidney, where a concentration of 0.011 mg/kg cyclaniliprole was found in one of the three animals. Concentrations of cyclaniliprole at the highest feeding level of 11.6 ppm were highest in liver (max 0.14 mg/kg) followed by fat (0.12 mg/kg), kidney (0.11 mg/kg), and muscle (0.032 mg/kg). Residues of NK-1375, NSY-27, and YT 1284 were either not detected or below the LOQ of < 0.01 mg/kg in all tissues and milk. Metabolite NSY-28 was found only in liver (11.6 ppm dose level) at 0.014–0.032 mg/kg and in kidney and subcutaneous fat at 0.014 mg/kg of one cow.

The results of the depuration group indicate that the total cyclaniliprole residues accumulate and slowly decline after the administration of the cyclaniliprole has stopped.

No laying hen study was submitted.

Animal commodities maximum residue level concentrations

Mammals

Dietary burden calculations demonstrate that the highest (0.12 ppm) and mean (0.066 ppm) dietary intake from beef and dairy cows is lower than the lowest intake levels (1.2 ppm) used in the dietary feeding study. Mean and highest residue levels observed in whole milk, muscle, liver and fat are either not detectable or below the LOQ of 0.01 mg/kg. At the 1.2 ppm feeding level of the feeding study some residues were observed in milk fat and kidney. These tissues were therefore considered for maximum residue and STMR calculation by extrapolation. The high and mean residues in the tissues and milk were calculated by extrapolating the maximum dietary burden (0.12 ppm) or median dietary

burden (0.066 ppm) from the relevant feeding level (1.2 ppm cyclaniliprole) from the dairy cow feeding study, using the highest or median tissue and milk (fat) concentrations.

	Feeding level (ppm) for milk residues	Residues (mg/kg) in milk cream [a]	Feed level (ppm) for tissue residues	Residues (mg/kg) in			
				Muscle	Liver	Kidney	Fat
MRL beef of dairy cattle							
Feeding study [b]	1.2	0.015	1.2	ND	< 0.01	0.011	< 0.01
Dietary burden and high residue estimate	0.12	0.0019	0.12	< 0.01	< 0.01	0.0011	< 0.01
STMR beef or dairy cattle							
Feeding study [c]	1.2	< 0.01	1.2	ND	< 0.01	0.010	< 0.01
Dietary burden and median residue estimate	0.066	0.0006	0.095	0	0.0008	0.0008	0 0008

ND = not detectable

[a] Residues were detected in milk cream only. No residues were found in skimmed milk. Based on the default fat content of 4%, the STMR for mammalian milk was estimated at 0.000024 mg/kg (0.04 × 0.0006 mg/kg)

[b] highest residue for tissues and mean residues for milk

[c] mean residues for tissues and mean residues for milk

The Meeting estimated maximum residue levels of 0.01* mg/kg for mammalian milk, meat (based on fat), liver, kidney and fat. Though cyclaniliprole is fat soluble, residues in milk cream in the dietary feeding study are at max 0.015 mg/kg at the 1.2 ppm feeding level, indicating that a maximum residue level for milk fat of 0.01* mg/kg is sufficient as well. The Meeting estimate an STMRs of 0.0006 mg/kg for mammalian milk fat and 0.0008 mg/kg for liver, kidney and fat. The STMR for mammalian muscle was set at 0 mg/kg. Based on the default fat content of 4%, the STMR for mammalian milk was estimated at 0.000024 mg/kg (0.04 × 0.0006 mg/kg).

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

The Meeting recommended the following residue definitions for cyclaniliprole.

For plants and animals: Definition of the residue for compliance with the MRL for plant and animal commodities: *cyclaniliprole.*

Definition of the residue for dietary risk assessment for plant commodities: *cyclaniliprole + 3-bromo-2-((2-bromo-4H- pyrazolo[1,5-d]pyrido[3,2-b]-[1,4]oxazin-4-ylidene)amino)-5-chloro-N-(1-cyclopropylethyl)benzamide (NK-1375), expressed as cyclaniliprole equivalents.*

The molecular weight conversion factor to express NK-1375 in cyclaniliprole equivalents = 1.064.

Definition of the residue for dietary risk assessment for animal commodities: *cyclaniliprole*

The Meeting considers the residue to be fat soluble.

CCN	Commodity name	Maximum residue levels (mg/kg)		STMR(-P) (mg/kg)	HR (P) (mg/kg)
		New	Previous		
FS 0013	Subgroup of Cherries (includes all commodities in this group)	0.9	-	0.17	-
VO 2700	Cherry Tomato	0.1	-	0.041	-
VC 2039	Subgroup of Cucumbers and Summer Squashes (includes all commodities in this group)	0.06	-	0.028	-
DV 0448	Tomato, dried	0.4	-	0.14	-

CCN	Commodity name	Maximum residue levels (mg/kg)		STMR(-P) (mg/kg)	HR (P) (mg/kg)
		New	Previous		
MO 0105	Edible offal (mammalian)	0.01*	-	kidney 0.0008 liver 0.0008	-
VO 2046	Subgroup of Eggplants (includes all commodities in this group)	0.1	-	0.041	-
VB 0042	Subgroup of Flowerhead Brassicas (includes all commodities in this group)	1	-	0.38	-
VB 2036	Subgroup of Head Brassicas (includes all commodities in this group)	0.7	-	0.066	-
VL 0054	Subgroup of Leaves of Brassicaceae *Brassica* spp. (includes all commodities in this group)	15	-	4.3	-
MM 0095	Meat (from mammals other than marine mammals)	0.01* (fat)	-	fat 0.0008 muscle 0	-
VC 2040	Subgroup of Melons, Pumpkins and Winter squashes (includes all commodities in this group)	0.15	-	0.055	-
MF 0100	Mammalian fats (except milk fats)	0.01*	-	0.0008	-
ML 0106	Milks	0.01*	-	0.000024	-
FM 0183	Milk fats	0.01*	-	0.0006	-
FS 2001	Subgroup of Peaches (including Apricots and Nectarines) (includes all commodities in this group)	0.3	-	0.0715	-
VO 0051	Subgroup of Peppers (excluding Martynia, Okra and Roselle)	0.2	-	0.063	-
HS 0444	Peppers, Chili, dried	2	-	0.63	-
FP 0009	Group of Pome fruits (includes all commodities in this group)	0.3	-	0.073	-
FS 0014	Subgroup of Plums (includes all commodities in this group)	0.2	-	0.067	-
DF 0014	Prunes, dried	0.8	-	0.25	-
FB 0269	Grapes	0.8	-	0.15	-
VO 0448	Tomato	0.1	-	0.041	-

na = not applicable, as there is no dietary burden for poultry.

Recommendations for dietary intake only

CCN	Commodity name	New MRL (mg/kg)	Previous MRL (mg/kg)	STMR-P (mg/kg)	HR-P (mg/kg)
JF 0226	Apple, juice	-	-	0.024	-
-	Grape, must	-	-	0.10	-
JF 0269	Grape, juice	-	-	0.54	-
-	Grape, wine	-	-	0.053	-
-	Tomato, canned	-	-	0.008	-
VW 0448	Tomato, paste	-	-	0.040	-
JF 0448	Tomato, juice	-	-	0.046	-

Recommendations for feed commodities for calculation of the dietary burdens.

CNN	Commodity	MRL (mg/kg)	Previous MRL (mg/kg)	Median residue -P (mg/kg)	Highest residue (mg/kg)
AS 0081	Straw and fodder, dry of cereal grains	0.45 (dw)	-	0.0475 (straw) [a]	0.18 (straw) [a]
AS 0081	Straw and fodder, dry of cereal grains	-	-	0.01 (forage) [a]	0.026 (forage) [a]
AB 0226	Apple pomace, wet (as received)	-	-	0.19	-
AB 0269	Grape pomace, wet (as received)	-	-	0.24	-
AB 0448	Tomato pomace, wet (as received)	-	-	0.020	-
AV 0480	Kale forage (leaves) – (based on mustard greens	-	-	4.0	6.5

CNN	Commodity	MRL (mg/kg)	Previous MRL (mg/kg)	Median residue -P (mg/kg)	Highest residue (mg/kg)
	dataset)				

dw dry weight

[a] Based on as received basis.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The current Meeting established an ADI of 0–0.04 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for cyclaniliprole were calculated for the 17 GEMS/Food cluster diets using STMRs estimated by the current Meeting for raw and processed commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3 of the 2017 Report.

The calculated IEDIs were 0–7% of the maximum ADI of 0.04 mg/kg bw.

The Meeting concluded that the long-term dietary exposure of residues to cyclaniliprole from uses considered by the current Meeting is unlikely to present a public health concern.

Short-term dietary exposure

The 2017 Meeting determined that establishment of an acute reference dose is unnecessary for cyclaniliprole. The Meeting therefore concluded that the short-term dietary exposure to residues of cyclaniliprole, resulting from uses that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
JSM0277	Airs, D.	2013	IKI-3106 and metabolites: Validation of methodology for the determination of residues of IKI-3106 and metabolites in animal tissues Huntingdon Life Sciences Ltd., UK; report no. JSM0277 GLP, unpublished
JSM0333	Alé, E.	2013a	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to broccoli in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0333 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0481	Alé, E.	2013b	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to broccoli in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0481 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0340	Alé, E.	2013c	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to Brussels sprouts in northern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0340 Ishihara Sangyo Kaisha, Ltd.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			GLP, unpublished
JSM0484	Alé, E.	2013d	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to Brussels sprouts in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0484 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0332	Alé, E.	2013e	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to cauliflowers in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0332 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0480	Alé, E.	2013f	IKI-3106 50SL (IBE 4064): Residue study (at harvest) with IKI-3106 50SL (IBE 4064) applied to cauliflowers in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0480 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0334	Alé, E.	2013g	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to head cabbage in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0334 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0482	Alé, E.	2013h	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to head cabbage in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0482 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0336	Alé, E.	2013i	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to outdoor sweet peppers in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0336 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0485	Alé, E.	2013j	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to outdoor sweet peppers in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0485 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0337	Alé, E.	2013k	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to indoor sweet peppers in northern and southern Europe in 2012

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Huntingdon Life Sciences, Ltd. Laboratory no. JSM0337 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0487	Alé, E.	2013l	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to indoor sweet peppers in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0487 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0335	Alé, E.	2013m	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to outdoor tomatoes in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0335 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0486	Alé, E.	2013n	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to outdoor tomatoes in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0486 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0353	Alé, E.	2013o	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to indoor tomatoes in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0353 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0354	Alé, E.	2013p	IKI-3106 50SL (IBE 4064): Residue study (at harvest, decline and processing) with IKI-3106 50SL (IBE 4064) applied to indoor tomatoes in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0354 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0488	Alé, E.	2013q	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to indoor tomatoes in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0488 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0483	Alé, E.	2013r	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to kale in northern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0483 Ishihara Sangyo Kaisha, Ltd.

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			GLP, unpublished
JSM0603	Alé. E.	2014	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to Brussels sprouts in southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0603 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0414	Bartolomé, J.	2013	IKI-3106 50SL (IBE 4064): Crop rotation residue study with IKI-3106 50SL (IBE 4064) applied to outdoor tomato and outdoor peppers in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0414 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0269	Brewin, S.	2012	IKI-3106 and NK-1375: Validation of Methodology for the Determination of Residues of IKI-3106 and NK-1375 in Grape, Wine, Peaches, Oilseed Rape Seed and Dry Beans Huntingdon Life Sciences Ltd., UK; report no. JSM0269 GLP, unpublished
JSM0542	Button, S.	2015	IKI-3106: Hydrolysis under Simulated Processing Conditions Huntingdon Life Sciences, Ltd. Laboratory no. JSM0542 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
	Cho, B. *et al.*	2013	Determination of residues for IKI-3106 4.5% SL in Red pepper Croen Research Inc., Korea unpublished
13510.6102	Connor, S.	2013	[^{14}C]IKI-3106 – Aerobic soil metabolism and degradation Smithers Viscient, USA; report no. 13510.6102 GLP, unpublished
JSM0053	Crowe, A.	2013a	IKI-3106: Metabolism in apples Huntingdon Life Sciences, Ltd. Laboratory no. JSM0053 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0054	Crowe, A.	2013b	IKI-3106: Metabolism in lettuce Huntingdon Life Sciences, Ltd. Laboratory no. JSM0054 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0055	Crowe, A.	2013c	IKI-3106: Metabolism in potatoes Huntingdon Life Sciences, Ltd. Laboratory no. JSM0055 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0148	Crowe, A.	2013d	IKI-3106: Accumulation in confined rotational crops Huntingdon Life Sciences, Ltd. Laboratory no. JSM0148 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
UPL-1113	Farrell, P.	2013a	Determination of residues of IKI-3106 and NK-1375 in pome fruit following two (2) applications of IKI-3106 50SL applied as a foliar spray at various timings before harvest Peracto Pty Ltd., Australia; report no. UPL-1113 GLP, unpublished
ISK12433	Farrell, P.	2013b	Determination of residues of IKI-3106 and NK-1375 in apples following two (2) applications of IKI-3106 50SL applied at various timings prior harvest Peracto Pty Ltd., Australia; report no. ISK12433

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			GLP, unpublished
JP2013C091	Hitoshi, I.	2013	IKI-3106 50SL Chinese Cabbage Residue Test Japan Plant Protection Association, Japan; report no. JP2013C091 GLP, unpublished
JSM0060	Jones, A.	2013	IKI-3106: Metabolism in laying hens Huntingdon Life Sciences, Ltd. Laboratory no. JSM0060 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0059	Kane, T.	2013	IKI-3106: Metabolism in the lactating goat Huntingdon Life Sciences, Ltd. Laboratory no. JSM0059 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JP2012C100	Kimikazu, S.	2013a	IKI-3106 50SL Grape Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2012C100 GLP, unpublished
JP2013C280	Kimikazu, S.	2013b	IKI-3106 50SL Grape Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2013C280 GLP, unpublished
JP2011C133	Koki, M.	2012	Crop Residue Study on Tea with IKI-3106 SL50 Japan Plant Protection Association, Japan; report no. JP2011C133 GLP, unpublished
JP2011C132	Kouji, N.	2012	IKI-3106 50SL Tomato Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2011C132 GLP, unpublished
no code	Kuzaki, I., and Naruto, T.	2013	Crop Residue Analysis Report Institute of Environmental Toxicology, Japan; Non-GLP, unpublished
IB-2013-JAM-002-01-01	McDonald, J.A. and Wiedmann, J.L.	2014a	Magnitude of Residues of IKI-3106 on grapes - USA & Canada in 2013 ISK Biosciences Corporation, USA; report no. IB-2013-JAM-002-01-01 GLP, unpublished
IB-2013-JAM-003-01-01	McDonald, J.A. and Wiedmann, J.L.	2014b	Magnitude of Residues of IKI-3106 on Cucurbits - USA & Canada in 2013 ISK Biosciences Corporation, USA; report no. IB-2013-JAM-003-01-01 GLP, unpublished
IB-2012-JAM-001-01-01	McDonald, J.A. and Wiedmann, J.L.	2014c	Magnitude of Residues of IKI-3106 on Lettuce and Spinach USA & Canada in 2012 ISK Biosciences Corporation, USA; report no. IB-2012-JAM-001-01-01 GLP, unpublished
13510.6103	McLaughlin, S. P.	2013	[^{14}C]IKI-3106 – Aerobic degradation in four soils Smithers Viscient, USA; report no. 13510.6103 GLP, unpublished
JSM0423	Miller, C.	2014	IKI-3106 and metabolite NK-1375: Storage stability in a range of crop matrices for periods of up to 18 months Huntingdon Life Sciences Ltd., UK; report no. JSM0423 GLP, unpublished
JSM0755	Miller, C.	2015	Cyclaniliprole & NK-1375: Independent laboratory validation of the methodology (AOAC official method 2007.01) for the determination of residues of Cyclaniliprole and it's metabolite (NK-1375) in food of plant origin (grape, peach, oil seed rape and dry bean) Huntingdon Life Sciences Ltd., UK; report no. JSM0755 GLP, unpublished
XR44SB	Miller, C.	2016a	BPQO, BCPBA and YT-1327: Validation of Methodology for the Determination of Residues in Tomato and Grape (RAC and Processed Fractions) Envigo CRS Limited, UK; report no. XR44SB

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			GLP, unpublished
BS38WY	Miller, C.	2016b	BPQO, BCPBA and YT-1327: Residue Study (at Harvest and processingXR44) With IKI-3106 50SL (IBE 4064) Applied to Wine grapes in Northern and Southern Europe Envigo CRS Limited, UK; report no BS38WY GLP, unpublished
IRA14028G	Nakano, I.	2014	Analytical method validation for the determination of Cyclaniliprole and its metabolite (NK-1375) in food of plant origin by QuEChERS Ishihara Sangyo Kaisha, Ltd., Japan; report no. IRA14028G GLP, unpublished
JSM0515	Ross, V.	2013	IKI-3106: Residue transfer study (feeding study) in dairy cows Huntingdon Life Sciences Ltd., UK; report no. JSM0515 GLP, unpublished
JSM0347	Schäufele, M.	2013a	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to apples in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0347 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0348	Schäufele, M.	2013b	IKI-3106 50SL (IBE 4064): Residue study (at harvest and processing) with IKI-3106 50SL (IBE 4064) applied to apples in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0348 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0473	Schäufele, M.	2013c	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to apples in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0473 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0338	Schäufele, M.	2013d	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to plums in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0338 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0476	Schäufele, M.	2013e	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to plums in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0476 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0329	Schäufele, M.	2013f	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to apricots in southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0329 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0474	Schäufele, M.	2013g	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to apricots in northern and southern Europe in 2013

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Huntingdon Life Sciences, Ltd. Laboratory no. JSM0474 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0351	Schäufele, M.	2013h	IKI-3106 50SL (IBE 4064): Residue study (at harvest) with IKI-3106 50SL (IBE 4064) applied to peaches in southern Europe in 2012 Huntingdon Life Sciences Ltd., UK; report no. JSM0351 GLP, unpublished
JSM0352	Schäufele, M.	2013i	IKI-3106 50SL (IBE 4064): Residue study (at harvest, decline and processing) with IKI-3106 50SL (IBE 4064) applied to peaches in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0352 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0475	Schäufele, M.	2013j	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to peaches in northern and southern Europe in 2013 Huntingdon Life Sciences Ltd., UK; report no. JSM0475 GLP, unpublished
JSM0330	Schäufele, M.	2013k	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to table grapes in southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0330 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0477	Schäufele, M.	2013l	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to table grapes in southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0477 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0349	Schäufele, M.	2013m	IKI-3106 50SL (IBE 4064): Residue study (decline) with IKI-3106 50SL (IBE 4064) applied to wine grapes in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0349 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0350	Schäufele, M.	2013n	IKI-3106 50SL (IBE 4064): Residue study (at harvest and processing) with IKI-3106 50SL (IBE 4064) applied to wine grapes in northern and southern Europe in 2012 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0350 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0478	Schäufele, M.	2013o	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to wine grapes in northern and southern Europe in 2013 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0478 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
JSM0667	Schäufele, M.	2014	IKI-3106 50SL (IBE 4064): Residue study (at harvest and decline) with IKI-3106 50SL (IBE 4064) applied to apricots in

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			northern Europe in 2014 Huntingdon Life Sciences, Ltd. Laboratory no. JSM0667 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
SQ74KP	Schäufele, M.	2016a	IKI-3106 50SL (IBE 4064): Residue Processing Study With Apples Spiked With IKI-3106 50SL (IBE 40664) Before Processing in 2015 Envigo CRS Limited, UK; report no. SQ74KP GLP, unpublished
QK27SS	Schäufele, M.	2016b	IKI-3106 50SL (IBE 4064): Residue Processing Study With Peaches Spiked With IKI-3106 50SL (IBE 40664) Before Processing in 2015 Envigo CRS Limited, UK; report no. QK27SS GLP, unpublished
HH97BD	Schäufele, M.	2016c	IKI-3106 50SL (IBE 4064): Residue Processing Study With Tomatoes Spiked With IKI-3106 50SL (IBE 40664) Before Processing in 2015 Envigo CRS Limited, UK; report no. HH97BD GLP, unpublished
120464	Schoenau, E.A.	2013	Independent Laboratory Validation of Ishihara Sangyo Kaisha (ISK) Residue Analytical Method for the Determination of IKI-3106 and its Metabolite NK-1375 in Almonds, Apples, Lettuce, and Wheat (Document Number: JSM0269) Golden Pacific Laboratories, USA; report no. 120464 GLP, unpublished
S13-03806	Schulz, D. and Herrig, A.	2013	Independent laboratory validation of the analytical method for the determination of IKI-3106 and metabolites in animal tissues Eurofins Agroscience Services Chem GmbH (EAS Chem), Germany; report no. S13-03806 GLP, unpublished
JP2011C362	Takashi, N.	2012a	IKI-3106 50SL Green Soybean Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2011C362 GLP, unpublished
JP2011C361	Takashi, N.	2012b	IKI-3106 50SL Soybean crop residue test Japan Plant Protection Association, Japan; report no. JP2011C361 GLP, unpublished
JSM0069	Turner, B.	2011	IKI-3106 (PAI): Water solubility Huntingdon Life Sciences, Ltd., UK; report no. JSM0069 GLP, unpublished
JSM0235	Turner, B.	2012a	IKI-3106 (PAI): Physico-chemical properties Huntingdon Life Sciences, Ltd., UK; report no. JSM0235 GLP, unpublished
JSM0241	Turner, B.	2012b	IKI-3106 (PAI): Vapour pressure and calculation of volatility (Henry's law constant) Huntingdon Life Sciences, Ltd., UK; report no. JSM0241 GLP, unpublished
JSM0267	Turner, B.	2012c	IKI-3106 (PAI): Partition coefficient Huntingdon Life Sciences, Ltd., UK; report no. JSM0267 GLP, unpublished
JSM0231	Turner, B.	2012d	IKI-3106 (PAI): Solvent solubility Huntingdon Life Sciences, Ltd., UK; report no. JSM0231 GLP, unpublished
JSM0242	Turner, B.	2012e	IKI-3106 (PAI): Thermal stability Huntingdon Life Sciences, Ltd., UK; report no. JSM0242 GLP, unpublished
JSM0228	Turner, B.	2012f	IKI-3106 (TGAI): Physico-chemical properties Huntingdon Life Sciences, Ltd., UK; report no. JSM0228 GLP, unpublished
JSM0232	Turner, B.	2012g	IKI-3106 (TGAI): Solvent solubility Huntingdon Life Sciences, Ltd., UK; report no. JSM0232 GLP, unpublished
JSM0505	Turner, B.	2013a	IKI-3106 (PAI): Partition coefficient by HPLC – effect of pH Huntingdon Life Sciences, Ltd., UK; report no. JSM0505 GLP, unpublished
JSM0504	Turner, B.	2013b	IKI-3106 (PAI): Water solubility – effect of pH

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
			Huntingdon Life Sciences, Ltd., UK; report no. JSM0504 GLP, unpublished
JSM0236	Turner, B.	2013c	IKI-3106 (PAI): Dissociation constant Huntingdon Life Sciences, Ltd., UK; report no. JSM0236 GLP, unpublished
JSM0230	Turner, B.	2013d	IKI-3106 (TGAI): Physico-chemical properties (EPA requirements) Huntingdon Life Sciences, Ltd., UK; report no. JSM0230 GLP, unpublished
JSM0051	Unsworth, R.H.	2010	IKI-3106: Hydrolysis in water Huntingdon Life Sciences, Ltd., UK; report no. JSM0051 GLP, unpublished
JSM0493	Unsworth, R.	2014a	IKI-3106: Radiovalidation of the Extraction Efficiency of the Residue Analytical Method for Animal Tissues Huntingdon Life Sciences Ltd., UK; report no. JSM0493 GLP, unpublished
JSM0492	Unsworth, R.	2014b	IKI-3106: Radiovalidation of the Extraction Efficiency of the Residue Analytical Method for Lettuce Plants Huntingdon Life Sciences Ltd., UK; report no. JSM0492 GLP, unpublished
IB-2012-JLW-022-01-01	Wiedmann, J.L., McDonald, J. A.	2013a	Field Accumulation of IKI-3106 in Rotational Crop Wheat - USA in 2012 Document Number: IB-2012-JLW-022-01-01 Ishihara Sangyo Kaisha, Ltd. GLP, unpublished
IB-2012-JLW-020-01-01	Wiedmann, J.L. and McDonald, J.A.	2013b	Magnitude of Residues of IKI-3106 on Apples - USA and Canada in 2012 ISK Biosciences Corporation, USA; report no. IB-2012-JLW-020-01-01 GLP, unpublished
IB-2013-JLW-005-01-01	Wiedmann, J.L. and McDonald, J.A.	2013c	Magnitude of Residues of IKI-3106 on Stone Fruit - USA and Canada in 2013 ISK Biosciences Corporation, USA; report no. IB-2013-JLW-005-01-01 GLP, unpublished
IB-2013-JLW-004-01-01	Wiedmann, J.L. and McDonald, J.A.	2014a	Magnitude of Residues of IKI-3106 on Pome Fruit - USA and Canada in 2013 ISK Biosciences Corporation, USA; report no. IB-2013-JLW-004-01-01 GLP, unpublished
IB-2012-JLW-028-01-01	Wiedmann, J.L. and McDonald, J.A.	2014b	Magnitude of Residues of IKI-3106 on Leafy Brassicas - USA and Canada in 2012 ISK Biosciences Corporation, USA; report no. IB-2012-JLW-028-01-01 GLP, unpublished
IB-2012-JLW-029-01-01	Wiedmann, J.L. and McDonald, J.A.	2014c	Magnitude of Residues of IKI-3106 on Fruiting Vegetables - USA and Canada in 2012 ISK Biosciences Corporation, USA; report no. IB-2012-JLW-029-01-01 GLP, unpublished
IB-2012-JLW-019-01-01	Wiedmann, J.L. and McDonald, J.A.	2014d	Magnitude of Residues of IKI-3106 on Almonds and Pecans - USA in 2012 ISK Biosciences Corporation, USA; report no. IB-2012-JLW-019-01-01 GLP, unpublished
JP2012C105	Yoshiyuki, T.	2013a	IKI-3106 50SL Cherry Tomato Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2012C105 GLP, unpublished
JP2012C106	Yoshiyuki, T.	2013b	IKI-3106 50SL Tomato Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2012C106 GLP, unpublished
JP2012C108	Yoshiyuki, T.	2013c	IKI-3106 50SL Chinese Cabbage Residue Test Japan Plant Protection Association, Japan; report no. JP2012C108 GLP, unpublished
JP2012C103	Yoshiyuki, T.	2013d	IKI-3106 50SL Green Soybean Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2012C103 GLP, unpublished

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JP2012C102	Yoshiyuki, T.	2013e	IKI-3106 50SL Soybean Crop Residue Test Japan Plant Protection Association, Japan; report no. JP2012C102 GLP, unpublished
JP2012C101	Yoshiyuki, T.	2013f	Crop Residue Study on Tea with IKI-3106 SL50 Japan Plant Protection Association, Japan; report no. JP2012C101 GLP, unpublished

REFERENCES SUBMITTED BUT NOT USED

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
JSM0279	Bull, A.	2012	NK-1375, a degradation product of IKI-3106: Acute oral toxicity to the rat (acute toxic class method) Huntingdon Life Sciences Ltd., UK; report no. JSM0279 GLP, unpublished
JSM0063	Button, S.	2011	IKI-3106: Soil photolysis Huntingdon Life Sciences Ltd., UK; report no. JSM0063 GLP, unpublished
JSM0278	May, K.	2012	NK-1375, a degradate of IKI-3106: Bacterial reverse mutation test, Huntingdon Life Sciences Ltd., UK; report no. JSM0278 GLP, unpublished